

*Ground Water**Engineering**Hydrocarbon**Remediation**Education*

July 10, 1991

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VIA FEDERAL EXPRESS

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Re: Torch Lake Superfund Site, Houghton County, Michigan

Subject: Final Work Plan for Drum Removal Effort

Dear Messrs. Nied and Felitti:

On behalf of Universal Oil Products Co., Inc., Quincy Mining Company, Quincy Development Corp., Houghton County Department of Public Works, Superior Crafts, Inc., and Rudolf Kump ("Respondents"), Geraghty & Miller, Inc. has developed and revised this Work Plan for the drum removal effort to take place at the Torch Lake Superfund Site in Houghton County, Michigan.

This Final Work Plan reflects the comments made by the OSC (Walter Nied) on the Draft Work Plan as relayed to us through Carol Grasner of the USEPA. Some editorial corrections (e.g., spelling errors, sentence structure, additional reference listings, etc.) have been incorporated into the Final Work Plan, as well; these editorial changes do not alter the substance of the Work Plan.

Geraghty and Miller has prepared this Final Work Plan as part of settlement negotiations to assist the Respondents with this proposed drum removal effort, and is looking forward to any additional questions or comments you may have regarding this Final Work Plan.

GERAGHTY & MILLER, INC.

Messrs. Nied and Felitti
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If you have any questions or comments regarding the information contained within the enclosed Final Work Plan, please do not hesitate to call Rich Bartelt or Gary Kruger at (312) 263-6703.

Sincerely,
GERAGHTY & MILLER, INC.



Gary W. Kruger
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enclosure

**FINAL WORK PLAN
TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN**

July 1991

Prepared For:

**Universal Oil Products Co, Inc.
Quincy Mining Company
Quincy Development Corp.
Houghton County Department of Public Works
Superior Crafts, Inc.
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1.0 INTRODUCTION

On behalf of Universal Oil Products Co., Inc., Quincy Mining Company, Quincy Development Corp., Houghton County Department of Public Works, Superior Crafts, Inc., and Rudolf Kump ("Respondents"), Geraghty & Miller, Inc. has developed this Work Plan for the drum removal effort to take place at the Torch Lake Superfund Site in Houghton County, Michigan. This Work Plan was developed in response to the United States Environmental Protection Agency (USEPA) administrative order by consent (the Order). The information provided herein is based on Geraghty & Miller's site inspections of May 1 and May 2, 1991; on a site inspection and field meeting with the USEPA On-Scene Coordinator (OSC), Walter Nied, conducted on May 22, 1991; and on oversight of test pit activities conducted during the week of June 3, 1991. Also considered in preparing this Work Plan is the information provided in the USEPA's Order, information provided during discussions with USEPA personnel, a viewing of the USEPA underwater videotape, review of a draft Action Memo, and the results of two previous drum sampling efforts conducted by USEPA subcontractors in June, 1989 and August, 1990. The approach outlined below was selected to provide protection of human health and the environment during drum characterization, staging, and removal activities while minimizing costs and being consistent with the NCP, the goals of the Order, and appropriate EPA guidance.

This Work Plan also includes a site health and safety plan (HASP), a sampling and analysis plan (SAAP), and a schedule of work to be performed. The HASP and the SAAP are attached to this report as Appendix A and Appendix B, respectively. The HASP was prepared in accordance with the Occupational Safety and Health Administration (OSHA) regulations applicable to Hazardous Waste Operations and Emergency Response, 29 CFR Part 1910. In addition, a dive plan will be prepared and attached as Appendix C to this Work Plan following the selection of an approved sub-contractor to perform the underwater

activities associated with the drum removal action. This dive plan will be modelled after the example dive plan that was provided by USEPA.

2.0 SITE BACKGROUND

2.1 DRUM SITE LOCATION AND HISTORY

Torch Lake is located in Michigan's Keweenaw Peninsula in Houghton County, Michigan (Figure 1). The lake has a surface area of 2717 acres, a mean depth of 56 feet, a maximum depth of 115 feet, and contains approximately 5.2×10^9 gallons of water (Donahue 1990). The Lake Superior district, in which Torch Lake resides, is the only place in the world where native (elemental) copper has been found in great abundance (Newton et al. 1942). For approximately 100 years, the lake was the site of copper milling and smelting activities, functioning as a source of milling water and a repository for the stampsands (also known as tailings) produced in the copper milling process. The stampsands consist primarily of natural rock that was crushed in the process used to separate the native copper from the associated rock material (Donahue 1990; Rose et al. 1986).

In 1968, a massive strike and market conditions forced the closing of the vast majority of the copper mines in the Torch Lake area, and work in the other mines ceased shortly thereafter. Since the closure of the mines, other business unrelated to copper smelting and milling operations have been conducted on or near the western shore of Torch Lake by others not parties to the Order.

In 1983, the Michigan Department of Public Health (MDPH) issued an advisory against the consumption of sauger and walleye from Torch Lake because of a high incidence of fish tumors noted on resident fish in studies conducted in the mid 1970s through the early 1980s. Although no human health effects were associated with the consumption of fish, the advisory was issued as a preventative measure until the causative factors of the fish tumors could be identified (Donahue 1990).

The MDPH fish advisory remains in effect although data collected during a 1988 fish sampling event demonstrated that the incidence of tumors have returned to normal, strongly suggesting that the liver tumor inducing agents no longer exist in the Torch Lake - Portage Lake fishery (Donahue 1990; MDNR 1989). In addition, a four-month tumor induction study using creosote and xanthate (two chemicals used in the copper separation process) failed to establish a causal relationship between the chemicals and liver abnormalities or fish tumors (Donahue 1990). Recent experiments have indicated that the skin tumors found in the fish at Torch Lake, which are common in walleyes from various waters in the Great Lakes basin and elsewhere, are likely to be viral in origin (Black 1989).

In 1984, the year after the fish advisory was issued, Torch Lake was proposed for listing on the National Priorities List (NPL) of toxic waste sites mainly because of the abnormally high incidence of fish tumors that were noted in previous studies (Rose et al. 1986). The site was officially placed on the NPL in June of 1988. The Remedial Investigation and Feasibility Study (RI/FS) process was initiated at the site in October, 1988 to assess any potential environmental concern on the site. The site was broken into three separate areas, called "Operable Units," with each unit to undergo a separate RI/FS. During the course of the RI for Operable Unit I (OU I), USEPA contractors identified a number of drums that are located in four specific on-land areas and one off-shore area (Figure 2) along the western shoreline of Torch Lake. The four relevant drum location areas (and the four areas addressed by this Work Plan) are:

- Area 1 - Old Calumet and Hecla smelting mill site near Lake Linden
- Area 2 - Ahmeek Mill site
- Area 3 - Tamarack site

- Area 4 - Quincy site

As a result of the discovery of drums, and after the subsequent sampling of the drums (see Section 2.3.3), the USEPA issued an administrative order on consent (the Order) to identify, sample, and remove those drums that are found to contain hazardous materials. This Work Plan was developed to satisfy the Order for the identification, sampling, and removal of hazardous drums located in the four areas noted above as well as the applicable adjacent off-shore areas on the western edge of Torch Lake.

2.2 DRUM SITE CHARACTERISTICS

The four areas where drums are located on-land in OU I are all a short distance from of the western shore of Torch Lake (Figure 2).

Area 1, the old Calumet and Hecla smelting mill site near Lake Linden, is characterized by the ruins of a former smelting facility. The foundations of several razed buildings remain throughout the area. Area 1 contains approximately 15 drums that are located on a steep loose brick and rubble slope that leads to the water line. The rubble slope is approximately 15 feet deep, and the drums appear to be limited to an area that runs approximately 40 feet along the embankment. The embankment should be able to be accessed by a backhoe or drum grapppler for drum removal, but a long reach will be required.

Area 2, located to the east of the former Ahmeek mill site, is also characterized by the ruins of several old mining buildings. The vast majority of the drums located in this area are slag drums that are located in an open area on the stampsands along the western edge of Torch Lake. These slag drums were sampled and characterized as non-hazardous by USEPA

contractors (Donahue 1990). The singular drum that was found to contain hazardous materials in this area was located on the edge of a construction debris pile. This drum does not appear to be associated with mining waste disposal practices, but appears to be a result of a more recent unauthorized disposal (Donahue 1990). All locations for drum sampling and removal from Area 2 should be accessible to necessary equipment.

Area 3, the Tamarack site, contains a wall of concrete rip rap and numerous slag filled drums set up as an engineered erosion protection barrier for the Soo Line railroad spur and State highway M-26. The erosion barrier is a steep wall of material approximately 150 feet long and 15 feet high, and currently serves to protect against erosion from the adjacent creek/floodway that leads into Torch Lake. Only portions of the wall are easily accessible to heavy equipment, but the wall is accessible by foot and can be easily inspected and sampled.

The Area 4 drum location consists of approximately four drums located in a wooded section of property approximately 30 feet from a gravel roadway. Some limited brush clearing may be necessary to facilitate any necessary overpacking and removal of drums from this area. The area should be accessible to heavy equipment, but backhoe mats may be necessary to operate in this low lying area.

The offshore areas of concern are typically characterized by 15% to 20% bottom slopes. The water offshore is relatively clear, but the fine nature of the silt that makes up the bottom sediment in some areas is easily disturbed, and may cause turbidity when bottom activity takes place.

2.3 PRIOR DRUM INVESTIGATIONS

Several drum investigations have been conducted on the Torch Lake site as part of the RI activities associated with OU I. These activities, which include on-land and offshore geophysical studies as well as several drum sampling events, are described below.

2.3.1 On-Land Geophysical Investigation

A site-wide on-land geophysical investigation has already been completed for the Remedial Investigation of the site. The purpose of the investigation was to locate suspected drum locations within the stampsands which were alleged to exist according to local rumors (Donahue 1989). The USEPA conducted magnetometry and ground penetrating radar (GPR) investigations on the stampsands during the week of May 8, 1989 to investigate these rumors.

The geophysical survey encompassed three separate areas in OU I and covered over 40 total acres of the site. The areas investigated included the Centerline Apartment site in Lake Linden, the Stampmill Site near the old Ahmeek Mill Site, and the Sewage Settlement Pond Site near Tamarack City.

The anomalies found during this investigation were investigated by the USEPA with the construction of a total of ten test pits in the three areas on site during the week of June 3, 1991. All ten test pits excavated in the areas showing anomalies revealed no buried drums or any other evidences of potentially hazardous materials located beneath the land surface.

2.3.2 Off-Shore Geophysical Investigation

A GPR survey and a subbottom profile (seismic) survey of the lake bottom was also conducted by the USEPA during the week of May 8, 1989. The area in which this survey was conducted is immediately off-shore from the Area 1 drum location. Technical difficulties and equipment limitations hampered both the marine GPR and the seismic survey (Donahue 1989), but several point targets (possibly drums) were located on the bottom of the lake (USEPA 1989). The existence of point targets on the lake bottom in this area is to be expected because of the concrete debris that is known to exist here. A remote operated vehicle (ROV) survey and follow-up dives in this area conducted by the USEPA in this area has confirmed the existence of an apparently limited number of submerged drums and other debris in this area. The USEPA has sampled one of the underwater drums during a dive on May 21, 1991, and the results from this sampling event are forthcoming.

2.3.3 Prior Drum Sampling Events

The USEPA has conducted two rounds of on-land sampling of the drums located in four distinct areas on the western shore of Torch Lake, and one round of drum sampling of a single underwater drum adjacent to Area 1. Analyses of the data made available to the Respondents from the USEPA drum sampling programs have indicated that of the 17 on-land drum samples collected to date, only one of these samples is classified as a RCRA hazardous waste. Four additional drum samples have indicated the presence of hazardous substances, but did not demonstrate the characteristics of a hazardous waste. The results from the underwater drum sampled are not yet available.

At least three of the 17 on-land samples collected were of drums that contained slag, and the results of the analyses performed on these samples have confirmed that the slag

material is not hazardous. The non-hazardous nature of the slag is significant, since the vast majority of all the drums found in the areas of concern are filled with slag. The existence of these slag-filled drums appears to be part of an engineered erosion protection system and not the result of waste drum deposition.

From a historical perspective, it was not a common practice to place molten slag from the copper smelting process into barrels or drums as a means of slag disposal. Empty barrels were worth money as a salvage material, and the much easier, more logical, and more cost effective means of granulating the slag and transporting it in bulk via a pump or railcar had been developed early in the mining process (Newton 1942). Any molten slag placed into drums or barrels would not have been done as a slag disposal technique, therefore, but as a means to create usable building material.

Preliminary results of a USEPA underwater investigation of the off-shore area near Area 1 (see Figure 2) indicated that drums are present below the water line in this area. The number or nature of the objects that appear to be drums in the under water videotape generated by the USEPA has not been accurately defined but EPA divers have confirmed the existence of underwater drums.

2.3.3.1 6/21/89 Drum Sampling Results

On June 21, 1989, Weston, Inc., a USEPA contractor, collected a total of eight drum samples from three areas on site. Three samples were collected from Area 1 along the shoreline, two samples were collected from drums in Area 2, and three samples were collected from Area 4 drums. One drum was analyzed for EP Toxicity metals, total and reactive cyanide, total and reactive sulfide, pH, and flashpoint; all other drum samples in this

round were analyzed for volatile organic compounds (VOCs), acid/base-neutral semivolatile organic compounds (ABNs), pesticides, PCBs, EP Toxicity metals, and Cyanide.

In general, only traces of volatile and semivolatile organics were found in this round of sampling (Donahue 1990), and none of the samples were considered a RCRA hazardous waste. The only significant detection of hazardous materials (4000 ppm of trichloroethylene) occurred in a drum containing greasy rags located in Area 2. This drum is not suspected to be the result of disposal associated with the mining activities of Calumet and Hecla, and more likely is the result of a recent unauthorized disposal (Donahue 1990).

In this first round of drum sampling, one drum of slag was sampled in Area 2. Only one drum of slag was sampled in this area according to the USEPA contractor because it was believed to be representative of all the slag drums (Weston 1990a). The slag was analyzed for EP Toxicity metals, total metals, volatile organic compounds, semi-volatile organic compounds, pesticides, PCBs, and cyanide. A review of the sampling results as presented in the Final Remedial Investigation (RI) Report for Operable Unit I revealed that the slag was clearly non-hazardous.

2.3.3.2 8/1/90 Drum Sampling Results

On August 1, 1990, Weston, Inc., a USEPA contractor, collected a total of nine drum samples from the four drum location areas on site. Four samples were collected from drums in Area 1 along the shoreline, one sample was collected from a drum in Area 2, two samples were collected from slag drums in Area 3, and two samples were collected from drums in Area 4. These samples were analyzed for flashpoint, TCLP metals, F-list solvents, VOCs, and ABNs.

The analytical results indicate that one of the drums sampled in Area 4 is a RCRA hazardous waste because it was found to contain VOCs that are F-listed hazardous wastes. One other drum in Area 4 was found to contain VOCs that were hazardous substances, as did two of the drums from Area 1 and the drum sampled from Area 2. The drum sampled in Area 2 during this round of sampling was the same drum sampled in the first round of sampling.

Area 3 slag drums were sampled for the first time during this round of sampling. These data from this round of sampling also confirm that the vitrified slag material is non-hazardous (Weston 1990b). Since the copper bearing rock smelted to produce the slag all came from the same general geographic region, the slag itself would be expected to be quite homogenous, and the slag found in one drum should be very similar to the slag found in another drum. The homogenous nature of the slag material found in the drums on the site are reflected by the consistently non-hazardous, low extractable metals content of the material tested from several different drums.

3.0 TASK DESCRIPTIONS

3.1 TASK 1 - CONTRACTOR PROCUREMENT

The Respondents have retained Geraghty & Miller, Inc. as a qualified contractor to undertake and complete the requirements of the Order. Specialized subcontractors to perform the drum removal tasks will be obtained following the approval of the Work Plan. In the event that the USEPA reasonably disapproves of a selected contractor, the Respondents will retain a different contractor to perform the work.

3.2 TASK 2 - TERRESTRIAL DRUM REMOVAL

The removal of drums on-land will be performed by following the general subtasks as listed below:

- 1) Visual Inspection
- 2) Removal
- 3) Initial Staging and Opening
- 4) Representative Sampling
- 5) Final Staging

A visual inspection will take place in the four on-land areas where drums are present. During this visual inspection, it can be determined what drums contain potentially hazardous materials. Drums located during this visual inspection will be initially segregated into potentially hazardous (e.g., tar-like semi-solid) and non-hazardous (e.g., wood, slag, and other obviously non-hazardous material) categories. The drums with potentially

hazardous contents will be subject to removal; drums that are RCRA empty or contain non-hazardous materials will remain in place.

The actual removal of the drums will be performed with a backhoe or crane with a drum sling, or with a backhoe using a drum grapppler attachment. If the initial inspection of the drums reveals that the integrity of the drum is breached and that the contents of the drum may spill out during removal, the drum will be fitted with an overpack as early in the removal process as possible. Any potentially hazardous drum that is leaking, badly corroded, or deformed will be overpacked or the contents will be transferred to a new or reconditioned drum. Consolidation of similar waste material to reduce the overall number of drums potentially requiring disposal may also take place at this time. All overpacks and drums will meet with DOT specifications with regard to waste-container compatibility, packaging, and labeling before being transported offsite.

Once the drums are removed, they will be held in an initial staging area where they are to be opened and sampled. Prior inspections of the drums on-site have revealed that the vast majority of the drums are already open and contain solid material. Those drums that contain materials that are not accessible to sampling may require opening. The drums will be opened using a non-sparking spike attached to a backhoe bucket.

Once the drums are opened, they will be visibly classified into different groups, and representative samples will be collected from the potentially hazardous drums. All samples of potentially hazardous substances will be analyzed for:

- Flash Point
- TCLP Metals
- Volatile Organic Compounds

- Semivolatile Organic Compounds

Details of the sampling and analysis protocol for drums can be found in the SAAP located in Appendix B.

Following the sampling procedure, any potentially hazardous drums will be staged in a secured area. The staging area will be constructed such that it will allow for the drums to be placed in rows two drums deep with eight feet between the rows for access of drum handling equipment.

The drums present in the four separate on-land locations each present unique situations that must be considered when implementing the removal action. The unique steps required for removal of drums from the four specific areas are outlined below.

3.2.1 Area 1 Drum Removal

There are drums present in Area 1 amongst the brick and rubble leading along a short section of shoreline in Torch Lake. The action taken in this area will consist of removing all visible drums on the embankment leading into Torch Lake. Any additional drums underneath the currently visible drums that are uncovered during the removal process will be removed, as well. This removal action will require the use of drum removal equipment with a relatively long reach. Removal of the underwater drums adjacent to Area 1 is addressed in Section 3.6 of this report.

3.2.2 Area 2 Drum Removal

In Area 2, the vast majority of drums present consist of barrels filled with solid slag, which is the vitrified rock material (gangue) that was melted along with the copper when the smelting of the copper bearing rock was performed (Gates 1969). It is apparent that the lines of slag-filled drums in this area were placed here as an engineered barrier to protect areas from water erosion due to the pumpage of large quantities of water and crushed rock back to Torch Lake from the rock crushing operation that occurred there.

The slag material from one drum on this site was sampled on July 21, 1989 by Weston, Inc., a USEPA contractor. Only one drum of slag was sampled in this area according to the USEPA contractor because it was believed to be representative of all the slag drums (Weston 1990a). A review of the sampling results as presented in the Final Remedial Investigation (RI) Report for Operable Unit I revealed that the slag was non-hazardous. To confirm the non-hazardous nature of the slag material found in the drums in Area 2, two additional drums of slag will be sampled following the procedures outlined in the SAAP. Since metals are the only potential contaminants of concern in the slag, the slag samples will be analyzed for TCLP Metals to confirm the non-hazardous nature of the material. If the slag is demonstrated to be toxic using the TCLP, the OSC will be consulted as to the appropriate course of action. If the samples confirm the previous tests, and the slag is found to be non-hazardous, all slag drums will remain in place.

A visual inspection of Area 2 will be conducted to determine if any anomalous non-slag drums contain potentially hazardous materials. Drums that are RCRA-empty, and drums that contain obviously nonhazardous materials (e.g., wood, concrete, etc.) will remain in-place. One drum in Area 2 has been sampled twice by a USEPA contractor (Weston, Inc.) and found to contain trichloroethylene; this drum will not be resampled as the contents

have already been characterized. Any other potentially hazardous drums will be staged, sampled, and analyzed as described above in Section 3.2 of this report.

3.2.3 Area 3 Drum Removal

As with Area 2, Area 3 contains a line of slag filled drums (along with some concrete rip rap and soil) that was placed for the benefit of the Soo Line Railroad and the State Highway Department as an engineered barrier to prevent water erosion of the adjacent Soo Line spur and State Road M-26. Two of the slag filled drums in this area were sampled on August 1, 1990 by Weston, Inc., a USEPA contractor. The data collected also confirm that the vitrified slag material is non-hazardous (Weston 1990b). To confirm the results of the USEPA contractor's sampling of the slag drums in this area, two additional drums of slag will be sampled and analyzed for TCLP metals. If the slag is demonstrated to be toxic using the TCLP, the OSC will be consulted as to the appropriate course of action. If the samples confirm the previous tests and the slag is found to be non-hazardous, all slag drums will remain in place.

An additional visual inspection of Area 3 will be conducted to determine if any anomalous non-slag drums exist in this area. Any potentially hazardous drums will be handled as described above in Section 3.2 of this report.

3.2.4 Area 4 Drum Removal

Area 4 contains one drum that was found to contain a hazardous waste (F-Listed solvents). This hazardous drum is located in a wooded area with several other drums. A hand truck or similar device may be used to transport these drums the small distance to the dirt roadway. As with the other areas, any potentially hazardous drums will be handled as described above in Section 3.2 of this report.

3.3 TASK 3 - CONTAMINATED SOILS REMOVAL

In all locations where potentially hazardous drums are removed from the surface, the spilled contents, if any, from potential hazardous drums will be placed into the container from which the substances were spilled. The soil in the immediate area of the drum will then be visually inspected and screened with an Organic Vapor Analyzer (OVA) or similar device to check the soil for the presence of volatile organic compounds. If the visual inspection and OVA screening result in an indication that the soils may be impacted with hazardous substances, additional amounts of soil will be removed and drummed until subsequent visual and OVA screenings reveal no such evidence of soil contamination, as agreed to by the OSC.

3.4 TASK 4 - OFFSHORE GEOPHYSICAL INVESTIGATION

A geophysical investigation of the areas offshore to a depth of 30 feet will be conducted using ground penetrating radar, magnetometry, side-scan sonar, or another method mutually agreed upon by the Respondents and the OSC. The purpose of the offshore geophysical survey is to locate any drums that may be present on the lake bottom adjacent to the shoreline. The potential areas of offshore geophysical investigation include the

western shore of Torch Lake between drum Area 1 and drum Area 4, to a maximum depth of 30 feet. Any anomalous areas that may contain drums will be further investigated through the use of a Remote Operated Vehicle (ROV) (see Task 5 below). The initial underwater geophysical survey will be conducted contiguous with the Respondents' property or current or former places of business operations in locations as agreed upon between the Respondents and the EPA. The specific areas of shoreline to be investigated will be determined after consultation with OSC.

3.5 TASK 5 - CONDUCT UNDERWATER SURVEY

If necessary, the Respondents will conduct a follow-up underwater survey using an ROV to confirm if any drums are located along the shoreline in Torch Lake to a depth of 30 feet contiguous to Respondents' property or current or former places of business operations. This survey will be performed following the identification of possible underwater drum locations using the geophysical method employed in Task 4.0. The ROV follow-up survey will be conducted only in those areas that are identified by the offshore geophysical investigation as potential drum location areas.

3.6 TASK 6 - UNDERWATER DRUM REMOVAL

All drums located during the underwater survey will be numbered and recorded in a log book. Those drums that are filled only with water, natural silt, slag, concrete, or other obviously non-hazardous materials will be photo-documented and left in place. The other drums that do not exclusively contain slag or other natural or non-hazardous material will be sampled underwater, if possible. Those drums that are closed or otherwise cannot be sampled under water will be overpacked underwater, removed, staged, and sampled on shore in the same manner as presented for Area 1 drums. Those drums that were sampled and are

found to contain hazardous materials will be overpacked underwater (using divers) and removed. Dive operators will conform to regulations for dive operations and will meet OSHA, EPA, and NOAA requirements.

3.7 TASK 7 - HAZARDOUS MATERIAL DISPOSAL

All materials containing hazardous wastes that are removed during this operation will be disposed of or treated at a facility approved by the OSC and in accordance with the Resource Conservation and Recovery Act of 1976 (RCRA), 42 U.S.C Section 6901, et seq., as amended, the U.S. EPA Revised Off-Site Policy, and all other applicable Federal, State, and local requirements.

3.8 TASK 8 - REPORTING REQUIREMENTS

A written progress report will be submitted to the OSC on a monthly basis. Each progress report will describe the actions taken to comply with the Order, including any results of sampling and analysis work, and will describe the significant work items planned for the following month.

In addition to the monthly reports, a final report will be drafted following the completion of the drum removal effort summarizing the actions taken to comply with the Order. The report will address the items listed in paragraph 24 of the Order. The final report will be submitted within 30 calendar days of completion of the work required by the USEPA.

4.0 SCHEDULE

The projected schedule of activities for the work included in this Work Plan is presented in Table 1. It is anticipated that the on-site work will commence in early August, 1991, following the approval of the Work Plan and subcontracting of specialized removal contractors.

It is important to note that this schedule makes certain specific logical assumptions regarding the intensity and duration of field work, and if actual field conditions vary from these assumptions, the schedule may be effected. Examples of situations that can effect the schedule include unusually inclement weather (including high winds for the underwater portion of the removal effort) or other Acts of God, labor disputes, and permitting difficulties.

The on-land removal and initial offshore investigations will be conducted concurrently in early August, followed by the follow-up offshore investigations and drum removal. After data is received back from the analytical laboratory in mid to late September, disposal of any hazardous materials can commence. It is anticipated that all on-site work will be completed by October 15, 1991, and the Final Report on the removal activities will be submitted by November 15, 1991.

5.0 REFERENCES

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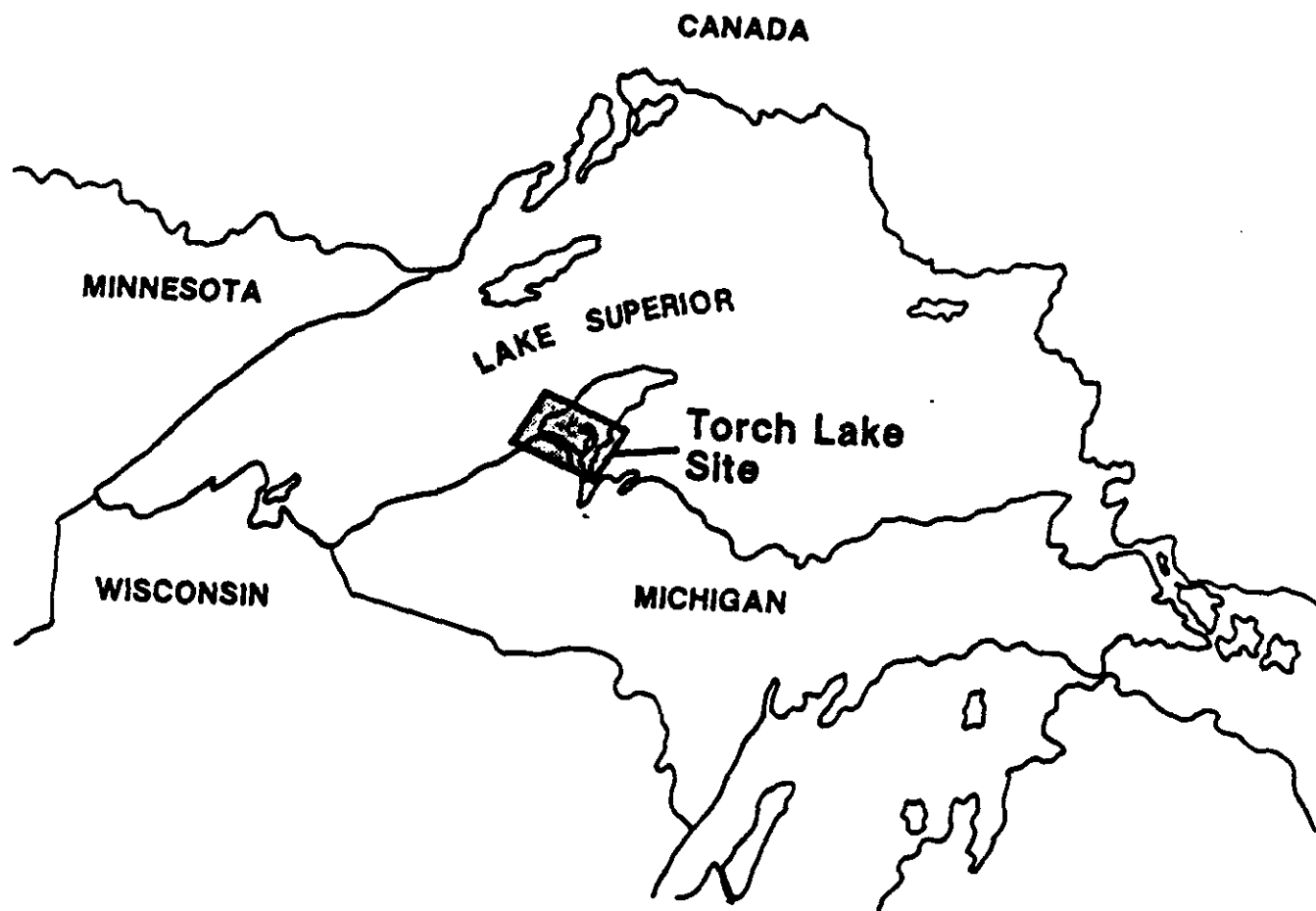
Weston 1990a. Site Assessment for Torch Lake, Houghton County, Michigan, February, 1990.

Weston 1990b. Torch Lake Drum Sampling Results Letter to Mr. Duane Heaton, USEPA, September 25, 1990.

¹ Copies of relevant portions of these references will be supplied to the USEPA on request.

Table 1: Drum Removal Schedule
Torch Lake Superfund Site, Houghton County, Michigan
Draft Work Plan

TASK	July	August	September	October	November
Task 1: Contractor Procurement	*****				
Task 2: Terrestrial Drum Removal		*****			
Task 3: Offshore Geophysical Investigation		****			
Task 4: Underwater Survey		****			
Task 5: Underwater Drum Removal		*****			
Task 6: Hazardous Materials Disposal				*****	
Task 8: Reporting Requirements	*	*	*	*	*



Source: Donohue, March 1989



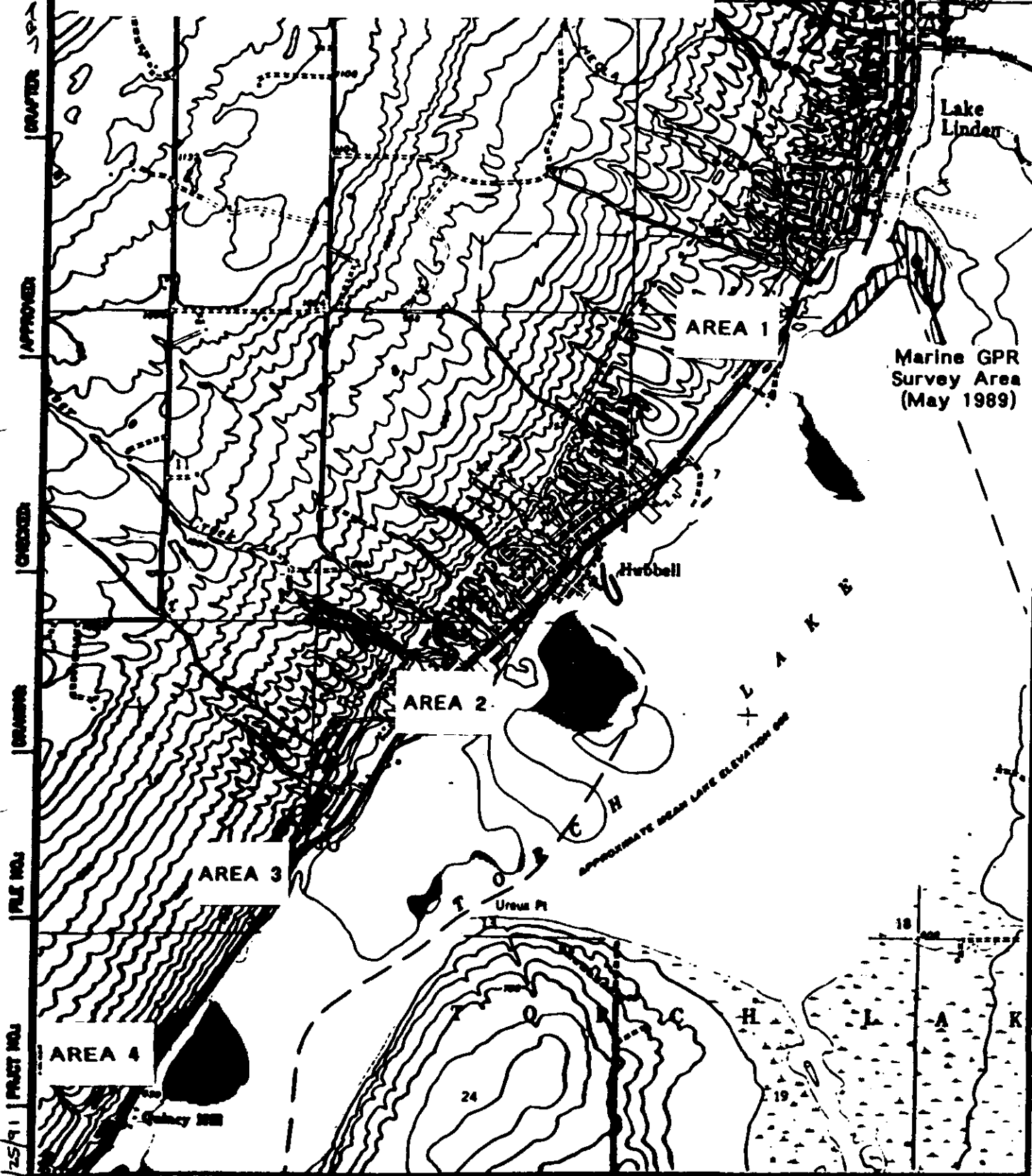
SITE LOCATION MAP

TORCH LAKE SUPERFUND SITE
HOUGHTON COUNTY, MICHIGAN

FIGURE

1

Source: USGS 7.5 Series Topographic Map, Laurium, MI



GERAGHTY & MILLER, INC.
Environmental Services

DRUM LOCATION AREAS
TORCH LAKE SUPERFUND SITE
HOUGHTON COUNTY, MICHIGAN

FIGURE
2

APPENDIX A
HEALTH AND SAFETY PLAN

**FINAL HEALTH AND SAFETY PLAN
TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN**

July 1991

Prepared For:

Universal Oil Products Co, Inc.
Quincy Mining Company
Quincy Development Corp.
Houghton County Department of Public Works
Superior Crafts, Inc.
Rudolf Kump

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1.0 INTRODUCTION

On behalf of Universal Oil Products Co., Inc., Quincy Mining Company, Quincy Development Corp., Houghton County Department of Public Works, Superior Crafts, Inc., and Rudolf Kump ("Respondents"), Geraghty & Miller, Inc. has developed this Health and Safety Plan (HASP) for the drum removal effort to take place at the Torch Lake Superfund Site in Houghton County, Michigan. This HASP is a supplement to the Torch Lake Drum Removal Work Plan ("Work Plan") developed in response to the United States Environmental Protection Agency (USEPA) administrative order by consent (the Order). Note that some sections of the Work Plan, specifically those regarding site history and background, have been repeated here for convenience.

The elements of the HASP are based upon requirements described in the Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities (October 1985) and the final (March 1989) Occupational Safety and Health Administration (OSHA) rules (29 CFR Part 1910). These guidelines have been supplemented by information obtained during site visits and previous sampling events. All reasonable precautions will be taken by Geraghty & Miller, Inc. and its subcontractors to ensure the safety and health of workers and the general public. All work will be performed in accordance with applicable federal, state, and local regulations and recommendations.

The objective of this health and safety plan is to assure that safe working conditions exist at the site. The safety organization and procedures have been established based on an analysis of potential hazards and personnel protection measures which have been selected in response to these risks.

The focus of this work plan is on health and safety issues regarding on-land drum

removal and sampling efforts. Health and safety issues specifically related to offshore activities will be addressed by the USEPA approved dive contractor, once selected.

2.0 GERAGHTY & MILLER, INC. RESPONSIBILITY

Geraghty & Miller, Inc. will be responsible for the adherence of its personnel to the HASP during the investigation and will ensure that all work is performed in accordance with the health and safety requirements described herein, the current edition of the OSHA rules for hazardous waste operations, and all appropriate federal, state, and local health and safety regulations. Subcontractors to Geraghty & Miller will submit and operate under their own health and safety plan.

Geraghty & Miller's Site Health and Safety Officer (HSO) will be determined prior to initiating field activities. The HSO's duties are to implement, monitor, and enforce the Site Health and Safety Plan. The HSO has the option to implement requirements in addition to those described herein on a case-by-case basis. Geraghty & Miller staff working on-site will be designated Health and Safety Coordinators (other employees to be designated later) and will enforce on-site adherence to the HASP and maintain communication with the HSO regarding site conditions and health and safety issues. Should an unforeseen or site-specific safety-related factor, hazard, or condition become evident during the field work, Geraghty & Miller will take action to reestablish safe working conditions and to safeguard site personnel, the public, and the environment. The site HSO will immediately contact Todd Udvig, who is Geraghty & Miller's Regional Health and Safety Officer, and update him on the current situation and alternatives.

3.0 SITE LOCATION AND DESCRIPTION

3.1 DRUM SITE LOCATION AND HISTORY

Torch Lake is located in Michigan's Keweenaw Peninsula in Houghton County, Michigan (Figure 1). The lake has a surface area of 2717 acres, a mean depth of 56 feet, a maximum depth of 115 feet, and contains approximately 5.2×10^9 gallons of water (Donahue 1990a). The Lake Superior district, in which Torch Lake resides, is the only place in the world where native (elemental) copper has been found in great abundance (Newton et al. 1942). For approximately 100 years, the lake was the site of copper milling and smelting activities, functioning as a source of milling water and a repository for the stampsands (also known as tailings) produced in the copper milling process. The stampsands consist primarily of natural rock that was crushed in the process used to separate the native copper from the associated rock material (Donahue 1990a; Rose et al. 1986).

In 1968, a massive strike and market conditions forced the closing of the vast majority of the copper mines in the Torch Lake area, and work in the other mines ceased shortly thereafter. Since the closure of the mines, other business unrelated to copper smelting and milling operations have been conducted on or near the western shore of Torch Lake by others not parties to the Order.

In 1983, the Michigan Department of Public Health (MDPH) issued an advisory against the consumption of sauger and walleye from Torch Lake because of a high incidence of fish tumors noted on resident fish in studies conducted in the mid 1970s through the early 1980s. Although no human health effects were associated with the consumption of fish, the advisory was issued as a preventative measure until the causative factors of the fish tumors could be identified (Donahue 1990a).

The MDPH fish advisory remains in effect although data collected during a 1988 fish sampling event demonstrated that the incidence of tumors have returned to normal, strongly suggesting that the liver tumor inducing agents no longer exist in the Torch Lake - Portage Lake fishery (Donahue 1990a; MDNR 1989). In addition, a four-month tumor induction study using creosote and xanthate (two chemicals used in the copper separation process) failed to establish a causal relationship between the chemicals and liver abnormalities or fish tumors (Donahue 1990a). Recent experiments have indicated that the skin tumors found in the fish at Torch Lake, which are common in walleyes from various waters in the Great Lakes basin and elsewhere, are likely to be viral in origin (Black 1989).

In 1984, the year after the fish advisory was issued, Torch Lake was proposed for listing on the National Priorities List (NPL) of toxic waste sites mainly because of the abnormally high incidence of fish tumors that were noted in previous studies (Rose et al. 1986). The site was officially placed on the NPL in June of 1988. The Remedial Investigation and Feasibility Study (RI/FS) process was initiated at the site in October, 1988 to assess any potential environmental concern on the site. The site was broken into three separate areas, called "Operable Units," with each unit to undergo a separate RI/FS. During the course of the RI for Operable Unit I (OU I), USEPA contractors identified a number of drums that are located in four specific on-land areas and one off-shore area (Figure 2) along the western shoreline of Torch Lake. The four relevant drum location areas (and the four areas addressed by this Work Plan) are:

- Area 1 - Old Calumet and Hecla smelting mill site near Lake Linden
- Area 2 - Ahmeek Mill site
- Area 3 - Tamarack site

- Area 4 - Quincy site

As a result of the discovery of drums, and after the subsequent sampling of the drums (see Section 2.3.3), the USEPA issued an administrative order on consent (the Order) to identify, sample, and remove those drums that are found to contain hazardous materials. This Work Plan was developed to satisfy the Order for the identification, sampling, and removal of hazardous drums located in the four areas noted above as well as the applicable adjacent off-shore areas on the western edge of Torch Lake.

3.2 DRUM SITE CHARACTERISTICS

The four areas where drums are located on-land in OU I are all a short distance from of the western shore of Torch Lake (Figure 2).

Area 1, the old Calumet and Hecla smelting mill site near Lake Linden, is characterized by the ruins of a former smelting facility. The foundations of several razed buildings remain throughout the area. Area 1 contains approximately 15 drums that are located on a steep loose brick and rubble slope that leads to the water line. The rubble slope is approximately 15 feet deep, and the drums appear to be limited to an area that runs approximately 40 feet along the embankment. The embankment should be able to be accessed by a backhoe or drum grapppler for drum removal, but a long reach will be required.

Area 2, located to the east of the former Ahmeek mill site, is also characterized by the ruins of several old mining buildings. The vast majority of the drums located in this area are slag drums that are located in an open area on the stampsands along the western edge of Torch Lake. These slag drums were sampled and characterized as non-hazardous by USEPA

contractors (Donahue 1990a). The singular drum that was found to contain hazardous materials in this area was located on the edge of a construction debris pile. This drum does not appear to be associated with mining waste disposal practices, but appears to be a result of a more recent unauthorized disposal (Donahue 1990a). All locations for drum sampling and removal from Area 2 should be accessible to necessary equipment.

Area 3, the Tamarack site, contains a wall of concrete rip rap and numerous slag filled drums set up as an engineered erosion protection barrier for the Soo Line railroad spur and State highway M-26. The erosion barrier is a steep wall of material approximately 150 feet long and 15 feet high, and currently serves to protect against erosion from the adjacent creek/floodway that leads into Torch Lake. Only portions of the wall are easily accessible to heavy equipment, but the wall is accessible by foot and can be easily inspected and sampled.

The Area 4 drum location consists of approximately four drums located in a wooded section of property approximately 30 feet from a gravel roadway. Some limited brush clearing may be necessary to facilitate the overpacking and removal of drums from this area. The area should be accessible to heavy equipment, but backhoe mats may be necessary to operate in this low lying area.

The offshore areas of concern are typically characterized by 15% to 20% bottom slopes. The water offshore is relatively clear, but the fine nature of the silt that makes up the bottom sediment in some areas is easily disturbed, and may cause turbidity when bottom activity takes place.

4.0 WASTE CHARACTERISTICS

The USEPA has conducted two rounds of on-land sampling of the drums located in four distinct areas on the western shore of Torch Lake, and one round of drum sampling of a single underwater drum adjacent to Area 1. Analyses of the data made available to the Respondents from the USEPA drum sampling programs have indicated that of the 17 on-land drum samples collected to date, only one of these samples is classified as a RCRA hazardous waste. Four additional drum samples have indicated the presence of hazardous substances, but did not demonstrate the characteristics of a hazardous waste. The results from the underwater drum sampled are not yet available.

At least three of the 17 on-land samples collected were of drums that contained slag, and the results of the analyses performed on these samples have confirmed that the slag material is not hazardous. The non-hazardous nature of the slag is significant, since the vast majority of all the drums found in the areas of concern are filled with slag. The existence of these slag-filled drums appears to be part of an engineered erosion protection system and not the result of waste drum deposition.

From a historical perspective, it was not a common practice to place molten slag from the copper smelting process into barrels or drums as a means of slag disposal. Empty barrels were worth money as a salvage material, and the much easier, more logical, and more cost effective means of granulating the slag and transporting it in bulk via a pump or railcar had been developed early in the mining process (Newton 1942). Any molten slag placed into drums or barrels would not have been done as a slag disposal technique, therefore, but as a means to create usable building material.

Preliminary results of a USEPA underwater investigation of the off-shore area near

Area 1 (see Figure 2) indicated that drums are present below the water line in this area. The number or nature of the objects that appear to be drums in the under water videotape generated by the USEPA has not been accurately defined but EPA divers have confirmed the existence of underwater drums.

4.1 6/21/89 DRUM SAMPLING RESULTS

On June 21, 1989, Weston, Inc., a USEPA contractor, collected a total of eight drum samples from three areas on site. Three samples were collected from Area 1 along the shoreline, two samples were collected from drums in Area 2, and three samples were collected from Area 4 drums. One drum was analyzed for EP Toxicity metals, total and reactive cyanide, total and reactive sulfide, pH, and flashpoint; all other drum samples in this round were analyzed for volatile organic compounds (VOCs), acid/base-neutral semivolatile organic compounds (ABNs), pesticides, PCBs, EP Toxicity metals, and Cyanide.

In general, only traces of volatile and semivolatile organics were found in this round of sampling (Donahue 1990a), and none of the samples were considered a RCRA hazardous waste. The only significant detection of hazardous materials (4000 ppm of trichloroethylene) occurred in a drum containing greasy rags located in Area 2. This drum is not suspected to be the result of disposal associated with the mining activities of Calumet and Hecla, and more likely is the result of a recent unauthorized disposal (Donahue 1990a).

In this first round of drum sampling, one drum of slag was sampled in Area 2. Only one drum of slag was sampled in this area according to the USEPA contractor because it was believed to be representative of all the slag drums (Weston 1990a). The slag was analyzed for EP Toxicity metals, total metals, volatile organic compounds, semi-volatile organic compounds, pesticides, PCBs, and cyanide. A review of the sampling results as presented

in the Final Remedial Investigation (RI) Report for Operable Unit I revealed that the slag was clearly non-hazardous.

4.2 8/1/90 DRUM SAMPLING RESULTS

On August 1, 1990, Weston, Inc., a USEPA contractor, collected a total of nine drum samples from the four drum location areas on site. Four samples were collected from drums in Area 1 along the shoreline, one sample was collected from a drum in Area 2, two samples were collected from slag drums in Area 3, and two samples were collected from drums in Area 4. These samples were analyzed for flashpoint, TCLP metals, F-list solvents, VOCs, and ABNs.

The analytical results indicate that one of the drums sampled in Area 4 is a RCRA hazardous waste because it was found to contain VOCs that are F-listed hazardous wastes. One other drum in Area 4 was found to contain VOCs that were hazardous substances, as did two of the drums from Area 1 and the drum sampled from Area 2. The drum sampled in Area 2 during this round of sampling was the same drum sampled in the first round of sampling.

Area 3 slag drums were sampled for the first time during this round of sampling. These data from this round of sampling also confirm that the vitrified slag material is non-hazardous (Weston 1990b). Since the copper bearing rock smelted to produce the slag all came from the same general geographic region, the slag itself would be expected to be quite homogenous, and the slag found in one drum should be very similar to the slag found in another drum. The homogenous nature of the slag material found in the drums on the site are reflected by the consistently non-hazardous, low extractable metals content of the material tested from several different drums.

5.0 POTENTIAL CONTAMINATION SOURCES AND HAZARD EVALUATION

Constant attention will be given to protecting on-site personnel from the physical and chemical hazards that may be encountered during the investigation. The field activities at the site will consist of on-land and offshore drum removal and drum sampling. The evaluation of potential hazards presented below is based on site history, numerous site inspections, and existing analytical data from previous drum sampling events.

The following potential chemical hazards may be encountered during field work at the site:

- Ingestion of contaminated waste (accidental/poor hygiene).
- Inhalation of contaminated particles, vapors or gases.
- Dermal contact with contaminated waste.
- Dermal contact with contaminated equipment and structures.

These hazards will be minimized by following the protocol for the designated working level of protection as described in Section 6.0, Personnel Protection Program.

Field personnel can also be exposed to a number of physical hazards during this project. Physical hazards that may be encountered are:

- Noise;
- Climbing hazards;
- Heat injury;
- Cold injury;
- Lacerations and contusions;

- Snake, insect and animal injuries; and
- Lifting hazards;
- Buried hazards.

General considerations for these common physical hazards are discussed below.

Noise Exposure

The field team may be exposed to excessive noise levels from removal equipment, although this is not anticipated. Hearing protection may be necessary during removal activities on the site.

Climbing Hazards

In the course of the removal activity, workers may have to work on equipment by climbing on the equipment, or may have to climb over inclines, mounds, etc. to attain access to some areas. The removal subcontractor will ensure that climbing activities will conform with any applicable NIOSH and OSHA requirements.

Heat Injury

During this project, workers may be required to wear protective clothing which insulates the body. A hazard may exist if workers wear protective clothing in temperatures over 70°F (see Section 6.4 of this report for details).

Cold Injury

Prolonged exposure to excessive cold or wet conditions may cause excessive loss of body heat (hypothermia) and/or frostbite (see Section 6.5. of this report for details).

Lacerations and Contusions (Cuts and Bruises)

The field team may cut or bruise themselves during this project. Removal activities usually involve contact with moving machinery and physical objects. The field personnel should be prepared to deal with cuts and bruises. A first aid kit will be present at the removal site.

Snake, Insect, and Animal Inflicted Injuries

The field team should be aware that site activities may disturb wildlife. Therefore, there is potential for field personnel to be bitten by snakes, animals, and insects. Prompt first aid measures are extremely important. All field team members should be properly briefed regarding the potential for encountering wildlife as well as prompt first aid procedures in the event of a snake, insect, or animal bite.

Lifting Hazards

Field team members may be exposed to injury caused by lifting heavy objects due to the fact that removal operations can involve manual movement of drums, overpacks, and various other pieces of equipment. All field team members should be trained in the proper method used to lift heavy equipment and cautioned against lifting objects that are too heavy for one person.

Buried Hazards

Whenever the ground is penetrated, the potential for cutting into buried hazards exists. A field representative should consult with local authorities about the location of underground utility lines (water, gas, electrical, telephone, cable TV, etc.), during the planning/mobilization phase.

Packaging and Shipping Hazards

All samples collected from the Torch Lake Superfund Site will be transported to a subcontracting laboratory(s) in compliance with Department of Transportation regulations. The instructions given below must be followed to comply with DOT regulations and reduce the risk of breakage in transportation.

- Package the primary container to protect it from breaking.
- Wrap the bottles with transparent tape and enclose bottles in a Zip-Loc™ (or equivalent) plastic bag.
- Classify and secure the shipping containers according to DOT regulations.

The primary physical hazard associated with conducting the investigation is working in the vicinity of the backhoe during removal of the drums. A minimum of Level D protection is required at the site (as described in Section 6.1) which includes a hard hat, safety glasses, and steel toed boots. Daily inspections of the backhoe will be conducted to check for worn hydraulic hoses and linkage pins. Caution will be exercised when moving and operating the backhoe. The working area around the backhoe will be considered an

exclusion zone and will be restricted to authorized personnel wearing the appropriate protective equipment.

6.0 PERSONNEL PROTECTION PROGRAM

A Personnel Protection Program has been established and will be maintained for all personnel working at the site. Geraghty & Miller will provide any necessary health and safety training to Geraghty & Miller personnel assigned to perform or oversee work, health and safety, security, administrative duties, or any other investigation-related functions at the site. Subcontractor safety meetings will be held before work begins each day and subcontractors will be trained in health and safety procedures for hazardous waste facilities. Separate protocol will be followed for site visitors as described in a later section.

6.1 LEVELS OF PERSONAL PROTECTION

Based on the concentrations of volatile organic compounds (VOCs) found in the drums during previous sampling events, the use of protective respiratory equipment will most likely be unnecessary for the majority of the activities onsite. The decision to upgrade or downgrade the protection levels will be the decision of the site HSO. Any respiratory protection plan implemented during on-site activities will be done in accordance with 29 CFR Part 1910.134.

In general, Level C personal protective equipment (PPE) has been selected for site drum removal and sampling of drums, but this level of protection may be downgraded to Level D or upgraded to Level B depending on results obtained from field analyses taken to monitor organic vapor levels (see Section 6.2). Initial sampling results obtained by USEPA contractors and the field observations made on the materials in the drums suggest that Level D PPE will be donned for the majority of all site activities.

Drum samples will be collected during the course of the removal activity as described

in the Work Plan and Sampling and Analysis Plan. Project personnel will take precautions to avoid dermal and inhalation exposure at all sample locations.

During sampling activities, smoking, chewing, or eating will be prohibited. Air monitoring equipment will be used to determine airborne quantities of VOCs. Surgical or nitrile gloves will be worn during all routine sampling tasks to avoid dermal contact with potentially hazardous materials.

When a drum of unknown contents is first approached for sampling, Level C PPE will be used until vapor monitoring justifies a downgrade to Level D. Due to the relatively low levels of VOCs found in the drums previously sampled by the USEPA contractor, Level C PPE will also be used during drum opening activities unless vapor monitoring justifies an increase in the PPE to Level B.

Sampling of the slag drums will be conducted in Level D PPE due to the non-volatile, non-hazardous nature of the slag material. An upgrade to Level C PPE will be made if air monitoring justifies and increase in the PPE level.

The levels of protection and the equipment utilized are defined as follows:

Level D: Tyvek coveralls.
 Hard hat.
 Gloves - nitrile or equivalent.
 Safety glasses.
 Steel-toed boots.
 Hearing protection (if needed)

- Level C:**
- Coveralls - Tyvek coveralls.
 - Full-face respirators with appropriate cartridges.
 - Hard hat.
 - Chemical-resistant, steel-toed and shanked boots.
 - Inner gloves - surgical type.
 - Outer gloves - nitrile or equivalent.
 - Outer boot covers.
 - Hearing protection (if needed).
 - Steel toes boots.
- Level B:**
- Coveralls - Tyvek coveralls.
 - Full-face positive pressure SCBA (self-contained breathing apparatus).
 - Hard hat.
 - Chemical-resistant, steel-toed and shanked boots.
 - Inner gloves - surgical type.
 - Outer gloves - nitrile or equivalent.
 - Outer boot covers.
 - Duct taping of boots and gloves to coveralls to eliminate possible leakage at the wrist and ankle.
 - Hearing protection (if needed).
 - Steel toed boots.

(No changes to the specified level of protection shall be made without the approval of the HSO.)

6.2 AIR MONITORING

Air quality in the breathing zone will be evaluated by collecting readings of organic vapor levels. Air monitoring readings will be collected after moving to each new removal area and whenever a drum is opened to examine and classify the contents or to collect samples. The HSO may alter this schedule as new information is obtained regarding health hazards at the site. Air monitoring equipment to be used for measurement of organic vapor levels will include, but are not limited to, an Organic Vapor Analyzer (OVA) or an HNu meter. The air in the vicinity of the drum opening will be surveyed as well as the air at shoulder height (breathing zone) in the vicinity of the working area. The air-monitoring equipment will be calibrated daily according to manufacturer instructions.

Level D protection will be used initially for setting up of the removal equipment and other activities not directly related to drum handling. If ambient air conditions exceed the background level of volatile organic compound (VOC) vapors for a sustained period of ten minutes or more, level D personnel protection will be considered insufficient and all work will cease and options evaluated including an upgrade to Level C or higher. The personnel protection level may be downgraded by the HSO to Level D when all monitoring parameters remain at or below background in the breathing zone for ten minutes or more.

Level C protection will be initially used during the drum removal process. If, during the drum removal process, ambient air conditions in the breathing zone exceed 5 ppm above the background level of VOC vapors, work will stop and alternatives for proceeding with the field work will be evaluated, including possible upgrade to Level B protection. The personnel protection level may be downgraded by the HSO to Level C when the VOC vapor level in the breathing zone drops below five ppm for ten minutes or more. Similarly, personnel protection can be downgraded to Level D by the HSO when the VOC vapor

concentration drops to background or below for ten minutes or more.

6.3 WORK ZONES

Prevention of exposure to and spread of contamination by activities at the site will be achieved through the establishment of work zones. Three work zones will be used: (1) Exclusion Zone, (2) Contaminant Reduction Zone, and (3) Support Zone. Flagging will be used to delineate these zones.

6.3.1 Exclusion Zone

The Exclusion Zone is the area where activities such as drum removal and sampling activities are conducted and where contaminants and physical hazards are potentially present. Only properly trained individuals who are wearing appropriate personal protection equipment will be allowed to enter and work in this zone. The size of the Exclusion Zone will be established by the HSO based on site-specific conditions.

6.3.2 Contaminant Reduction Zone

The Contaminant Reduction Zone is a corridor which leads from the Exclusion Zone to the Support Zone. This corridor will contain wash buckets, solid waste disposal containers, brushes, and equipment drop tarps. All decontamination activities will occur in this area.

6.3.3 Support Zone

The Support Zone is the area where the field team will be when not performing site

work. This area is to be used for rest breaks, eating, equipment storage, and staging. This zone will be located in an area that is known to be free of contamination and as far upwind from the actual removal activity site as practical.

6.4 HEAT STRESS CONTROL AND MONITORING

The HSO, in coordination with the backhoe supervisor, will set work and break schedules depending on how heavy the work load is and the outside temperature. Generally, workers conducting drilling activity in protective clothing need to break in the shade at least 10 minutes out of every hour during elevated temperatures (greater than 70° F). Rest time should also include fluid replacement with electrolytes.

During conditions where the temperature, humidity, and solar radiation are high and the air movement is low, the following procedures are recommended to prevent heat stress injury:

- Limit work activity periods to reduce the amount of heat the body produces.
- Workloads and/or duration of physical exertion should be less during the first days of exposure to heat and should be gradually increased to allow acclimatization.
- Schedule heavy work during the cooler periods of the day (e.g., early working).
- Alternate work and rest periods in heat stress conditions; in moderately hot conditions, 5-minute rest periods in the shade alternating with 25-minute work

periods in the sun may be desirable. Under severe conditions, the duration of rest periods should be increased.

- A heat stressed worker may lose up to a quart of water per hour. This loss must be replaced, or a rapid rise in body temperature will occur.

At the HSO's discretion, monitoring activities for heat stress will be performed when workers are using protective clothing in elevated temperatures. Recommended heat stress monitoring procedures include:

- Measurement of worker heart rate (number of pulse beats in 15 seconds x 4, or in 30 seconds x 2);
- Measurement of internal body temperature with dermal strip thermometers; and
- Observation of the field team for signs and symptoms of heat stress which include:
 - pale, clammy skin progressing to hot, dry and red skin,
 - profuse perspiration,
 - cramps,
 - headaches,
 - nausea, and
 - fainting.

Heat stress monitoring should be done at the HSO's discretion when temperatures are high or workers exhibit any indication of heat stress. In general, rest breaks will be taken at the worker's discretion and it is at these times that fluids will be replaced and heart rate and body temperatures will be measured and recorded in a logbook.

The following criteria will be used to institute heat stress controls (increase rest breaks, stop work, etc):

- Heart rate > 110 bpm (beats per minute) at beginning of rest period; shorten next work cycle by one-third;
- Heart rate > 90 bpm at 3 minutes into rest period; shorten next work cycle by one-third;
- Internal temperature > 99.6°F at beginning of rest period; shorten next work cycle by one-third; and
- Internal temperature > 100.6°F at any time; remove impermeable clothing and begin rest period until temperature drops to 99.6°F.

6.5 COLD INJURY CONTROL AND MONITORING

Temperatures in the upper peninsula of Michigan can reach very low levels from the late fall to early spring. Field personnel should be especially alert for cold injuries which are most likely to occur then an unprepared individual is exposed to cold temperatures. Temperature, humidity, precipitation, and wind all play a role in the development of cold injuries. Hypothermia may occur from exposure to temperatures above freezing, especially

from immersion in cold water or from the effect of wind. The first symptom is shivering, which is an attempt by the body to generate heat. Pulses become faint or difficult to detect. The body temperature will start to drop. Individuals may become drowsy and slow mentally, but for the most part they feel generally oriented and have no marked mental impairment. Their ability to move may be hampered and stiff and uncoordinated. Speech may become slurred. As body temperature drops further, shock becomes evident and the eyes become glassy, breathing becomes slow and shallow.

Another cold weather hazard is frost bite. The onset of frostbite starts with a sudden discoloration of the skin of the nose, ears, cheeks, fingers and/or toes; followed by a tingling sensation for a short time. Frostbite is a tissue injury caused by exposure to temperatures usually below 32° F, depending on the wind chill factor, duration of exposure, or adequacy of protection. When face, hands or feet stop hurting, frostbite has probably occurred. Superficial frostbite can be detected only after warming. Deep frostbite (below the skin layer), occurs most seriously in the feet and less commonly in the hands and face. With deep frostbite, gangrene can occur if the individual does not receive proper medical treatment.

The face needs extra protection in high winds. Use the "buddy system" to watch each other's face to detect frostbite early. Fingers and toes should be examined to maintain circulation and detect numbness. Do not touch cold metal objects with bare skin. Avoid excessive perspiration and replace damp/wet clothing immediately.

If cold injury occurs, get the victim medical attention as soon as possible. In the interim:

- Provide shelter from wind and administer warm fluids;

- Cover frozen areas with additional clothing or blankets;
- Do not rub or use direct heat or warm/hot water on the frostbitten area(s);
- Encourage gradual, gentle movement, but do not allow the person to walk if the feet are frozen; and
- Do not put frostbitten areas under warm or hot water.

6.6 TRAINING

Geraghty & Miller employees attend a 40-hour course on safety at chemical plants and hazardous waste sites. This course satisfies the initial training requirements of 29 CFR 1910.120 (OSHA regulation of hazardous waste site activities). In general, Geraghty & Miller personnel have been trained by the Red Cross for either basic first aid or cardiopulmonary resuscitation (CPR). Employees have also been certified by a physician to wear respirators.

Prior to initiating site work, all site personnel (Geraghty & Miller, Inc. and subcontractors) will be required to attend an orientation session given by the Health and Safety Coordinator. This session will take place at the site prior to the start of work and will include, but is not limited to, the following topics:

- Site history.
- Scope of field work.
- Specific hazards (toxicological data heat stress/exposure, other physical hazards).
- Hazard recognition.
- Standard operation procedures, including no smoking and no hand-to-mouth

- contact within the exclusion zones or prior to completing decontamination.
- Decontamination (personnel and equipment).
- Emergency procedures.
- Potential respirator use.

6.7 HEALTH MONITORING

Geraghty & Miller has established a Health Monitoring Program with occupational health specialists Carnow, Conibear and Associates, LTD in Chicago, Illinois. Geraghty & Miller employees receive yearly physicals consisting of the following:

- Personal, family and environmental history
- Hands-on physical examination
- Snellen's eye examination
- Hemocult testing (over age 40)
- Pulmonary Function Test
- Audiometric testing
- EKG
- Laboratory testing

A. Complete Blood Count

- 1) Red blood count
- 2) White blood count
- 3) Differential screening
- 4) Hemoglobin
- 5) Hematocrit

B. Urinalysis

- 1) Sugar
- 2) Albumin
- 3) Specific gravity
- 4) Microscopic

C. Laboratory Chemistries

- 1) A.G. ratio
- 2) Albumin
- 3) Alkaline, phosphatase
- 4) Bilirubin, total
- 5) Calcium
- 6) Chloride
- 7) Cholesterol
- 8) Creatinine
- 9) GGT
- 10) Globulin
- 11) Glucose
- 12) Iron
- 13) Lactic Dehydrogenase (LDH)
- 14) Phosphorus
- 15) Potassium
- 16) Protein, total
- 17) SGOT
- 18) SGPT
- 19) Sodium
- 20) Triglycerides
- 21) Urea nitrogen (BUN)
- 22) Uric acid
- 23) Blood lead level
- 24) Cadmium
- 25) Mercury
- 26) Zinc protoporphyrin

D. Special Testing

- 1) PCBs in serum

7.0 GENERAL WORK RULES

Field work will be conducted only during daylight hours unless adequate lighting is provided. The "buddy" system will be observed at all time when site personnel are required to wear respiratory protection.

Entry and exit into the continuous work area, exclusion zones, and contamination reduction zone will be permitted only through designated access points, except during an emergency or as authorized by the HSO. Personnel entering the exclusion zone must be wearing the required minimum protective clothing as specified in Section 6.0.

Eating, drinking, chewing gum or tobacco, smoking, or any practice that increases the probability of hand-to-mouth transfer and ingestion of material is prohibited in any area designated contaminated.

Hands and face must be thoroughly washed upon leaving the work area and before eating, or drinking. The entire body should be thoroughly washed as soon as possible after leaving the site.

No excessive facial hair, which interferes with a satisfactory fit of the mask-to-face seal, is allowed on personnel required to wear respiratory protective equipment. The HSO will determine if facial hair presents such an interference.

Contact with contaminated or suspected contaminated surfaces should be avoided. Personnel will be instructed not to walk through puddles, mud, and other discolored surfaces; lean, sit or place equipment on drums, containers, vehicles, or the ground, unless necessary.

All personnel assigned for on-site activities must be adequately trained and thoroughly briefed on anticipated hazards, equipment to be worn, safety practices to be followed, emergency procedures, and communications.

Daily safety meetings will be held with all field personnel prior to the start of work.

8.0 SITE VISITOR PROTECTION

Visitors to the site will be instructed to stay outside of the exclusion zone and remain within the support zone during the extent of their stay. Visitors will be cautioned to avoid skin contact with contaminated or suspected contaminated surfaces. During visitation, hand-to-mouth transfers will be reduced with special precautions not to eat, drink, smoke, or chew gum or tobacco. The use of alcohol prior to or during site visitation is prohibited. Authorized visitors on medication should request prior approval from the acting HSO before entering the site.

Authorized visitors requiring observation of the work in the exclusion zone must read the HASP and sign a form (Figure 3) stating that they have read and understand the safety protocol and will abide by it. All visitors entering the exclusion zone must wear appropriate personal protective gear. Should respiratory protective devices be necessary (Level C), visitors who require entrance to the exclusion zone must produce evidence that they have had a complete physical examination, have received respiratory protection training, and have been certified by a physician to use a respirator. Geraghty & Miller does not supply respirators or respirator cartridges to non-Geraghty & Miller personnel. Visitor inspection of the exclusion zone will be left to the discretion of the on-site HSO. No site visitors will be allowed to enter the drilling area exclusion zone if drilling crews and supervisory personnel are outfitted in Level B protection gear. Unauthorized visitation during Level B work will result in an immediate shutdown of drilling or well installation until the visitor leaves the exclusion zone.

9.0 EMERGENCY PROCEDURES

The following standard emergency procedures will be used by on-site personnel. The HSO will be notified of any on-site emergencies and be responsible for ensuring that the appropriate procedures are followed. An emergency report (Figure 4) must be completed and submitted to the site HSO for each instance of employee injury or possible exposure.

9.1 EMERGENCY PHONE NUMBERS AND HOSPITAL LOCATION

Emergency phone numbers will be posted at a conspicuous place in the support zone (Table 1). Directions to Portage View Hospital are given in Table 1 and a map with the route to the hospital is on Figure 5. The HSO will be responsible for making sure that all field personnel are familiar with the location of the hospital, and know where the emergency phone list and direction to the hospital are located.

9.2 PERSONNEL INJURY IN THE EXCLUSION ZONE

In the event of an injury in the exclusion zone, all site personnel will assemble at the decontamination line. The site Health and Safety Coordinator will evaluate the nature of the injury and the affected person will be decontaminated to the extent possible prior to movement to the support zone. Appropriate first aid will be initiated, and contact will be made with Portage View Hospital for an ambulance (if required). No person will reenter the exclusion zone until the cause of injury or symptoms are determined. An eye wash unit will be provided in the exclusion zone at the working area.

9.3 PERSONNEL INJURY IN THE SUPPORT ZONE

Upon notification of an injury in the support zone, the Project Manager, Health and Safety Officer, and Health and Safety Coordinator will assess the nature of the injury. If the cause of the injury or loss of the injured person does not affect the performance of site personnel, operations may continue, and the appropriate first aid and necessary follow-up, as stated above, should be initiated. First aid kits will be kept in appropriate places on the work site. The HSO will be responsible for making sure all personnel are familiar with the first aid kit locations. The HSO will also be responsible for the maintenance of the first aid kits.

9.4 FIRE/EXPLOSION EMERGENCY PROCEDURES

Fire hazards may exist in the following activities:

- Equipment refueling;
- High pressure water cleaning, fuel storage, and refueling activities;
- Any welding activities; and
- Decontamination with solvents.

The HSO will check to see that each vehicle and drilling rig fire extinguisher is appropriate for the fire hazard presented by this project. Generally, Type A, B, and C extinguishes are appropriate.

The field team will be prepared to fight small fires with extinguishes. In the event of a large fire, the field team will contact the appropriate authorities and report the fire.

The HSO will take the following action in the event of a fire:

- Notification of all site personnel and appropriate authorities that a fire exists;
- Shutdown site activities;
- Account for all site workers; and
- Evacuate the site, if necessary.

9.5 PERSONAL PROTECTIVE EQUIPMENT FAILURE

All personnel will work with a partner in the exclusion zone. If any site worker experiences a failure or alteration of protective equipment that affects the protection factor, that person and his/her partner will immediately leave the exclusion zone. Reentry will not be permitted until the equipment has been repaired/replaced.

10.0 REFERENCES

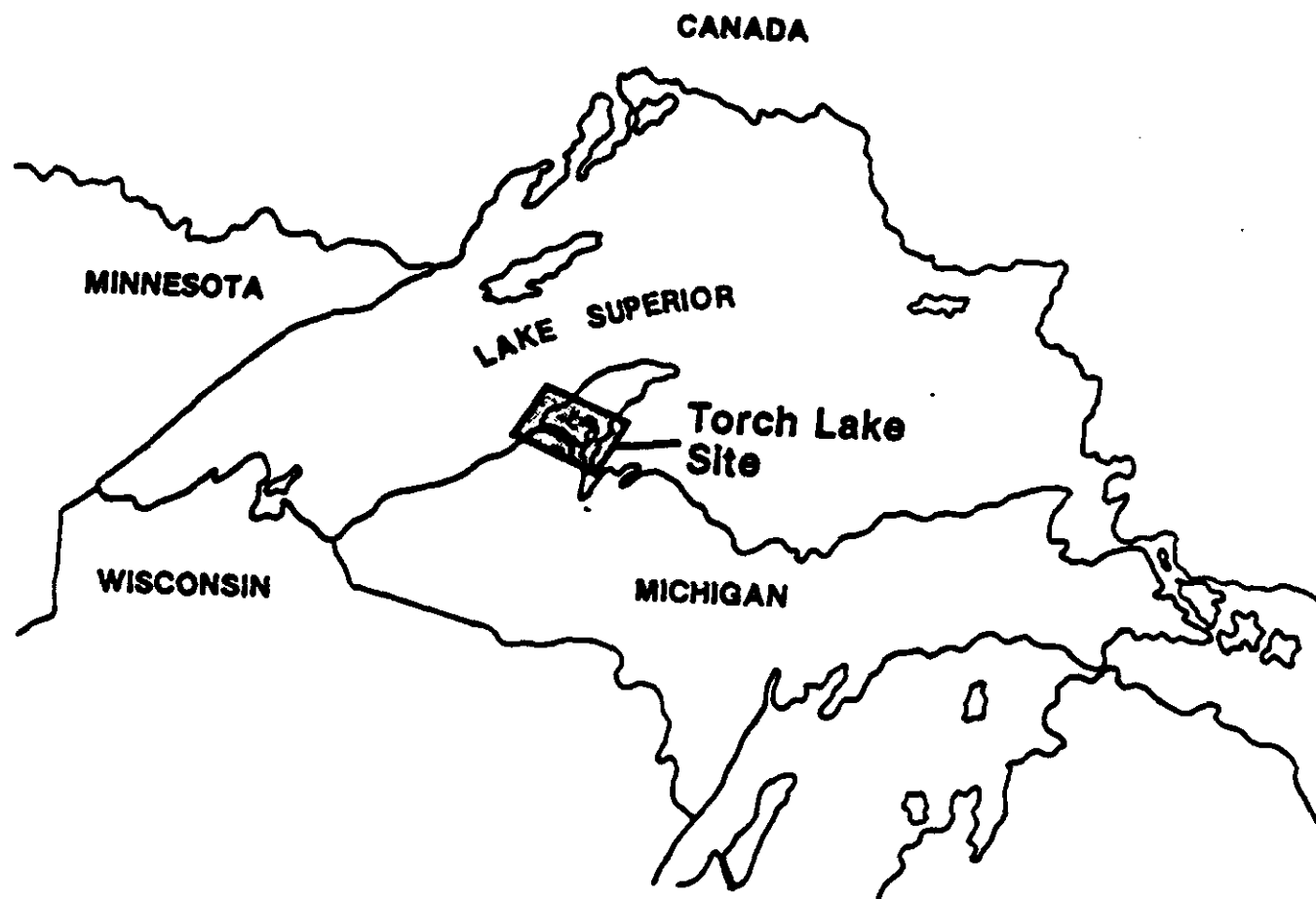
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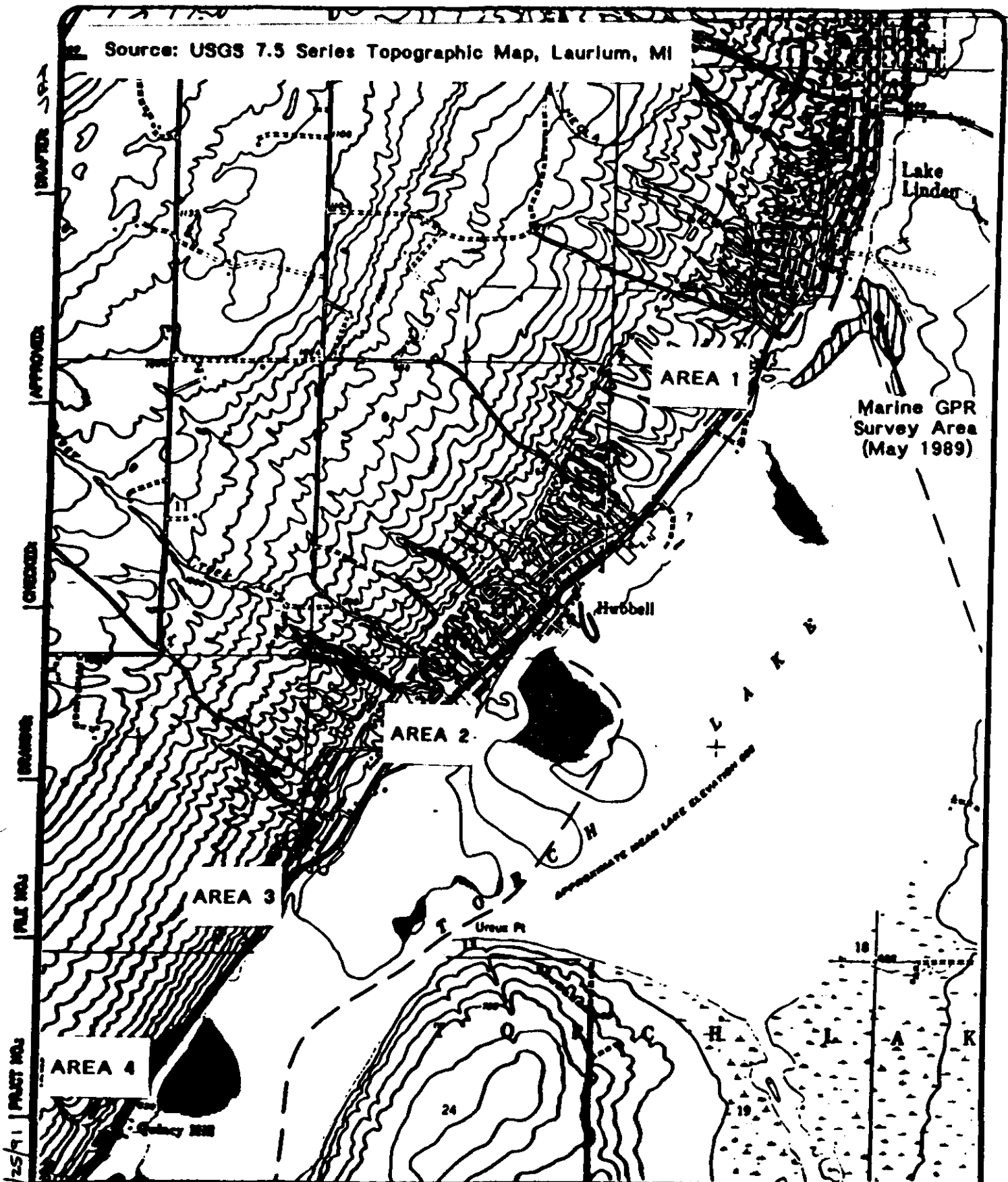
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¹ Copies of relevant portions of these references will be supplied to the USEPA on request.



Source: Donohue, March 1989

Source: USGS 7.5 Series Topographic Map, Laurium, MI



**GERAGHTY
& MILLER, INC.**
Environmental Services

DRUM LOCATION AREAS

TORCH LAKE SUPERFUND SITE
HOUGHTON COUNTY, MICHIGAN

FIGURE

2

**VISITOR REVIEW OF
SITE HEALTH AND SAFETY PLAN
TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN**

THE UNDERSIGNED VISITORS OF THE KOHLER COMPANY LANDFILL REQUIRE ENTRANCE TO THE EXCLUSION ZONE AND HAVE THOROUGHLY READ THE HEALTH AND SAFETY PLANS, UNDERSTAND THE POTENTIAL HAZARDS AT THE SITE AND THE PROCEDURES TO MINIMIZE EXPOSURE TO THE HAZARDS, WILL FOLLOW THE DIRECTION OF THE SITE HEALTH AND SAFETY OFFICER, AND WILL ABIDE BY THE HEALTH AND SAFETY PLAN.

[illegible]

FIGURE 4
EMERGENCY REPORT FORM
TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN

1. DATE _____
2. TIME OF ACCIDENT _____
CLIMATIC CONDITIONS _____
3. ON-SITE COORDINATOR _____
4. EMPLOYEE INJURED _____
5. COMPANY AFFILIATION _____
6. SOCIAL SECURITY NUMBER _____
7. INSURANCE COMPANY _____
8. NUMBER OF WORKERS AT SITE _____

NAMES OF WORKERS	COMPANY AFFILIATION
A. _____	_____
B. _____	_____
C. _____	_____
D. _____	_____
E. _____	_____
9. CIRCUMSTANCES OF THE INJURY/EMERGENCY ACTION _____

10. EMERGENCY ACTIONS TAKEN _____

11. WHAT FIRST AID WAS PROVIDED? _____

12. WAS AN EMERGENCY PHONE CALL MADE TO THE PROJECT SAFETY OFFICER? _____
IF SO, TIME: _____
13. AMBULANCE SERVICE USED _____
14. HOSPITAL USED _____
15. ATTENDING PHYSICIAN _____
16. COMPANY REPRESENTATIVES CONTACTED _____
17. CONTRACTOR REPRESENTATIVES CONTACTED _____

DWG NO: 06/25/91 PROJECT NO:

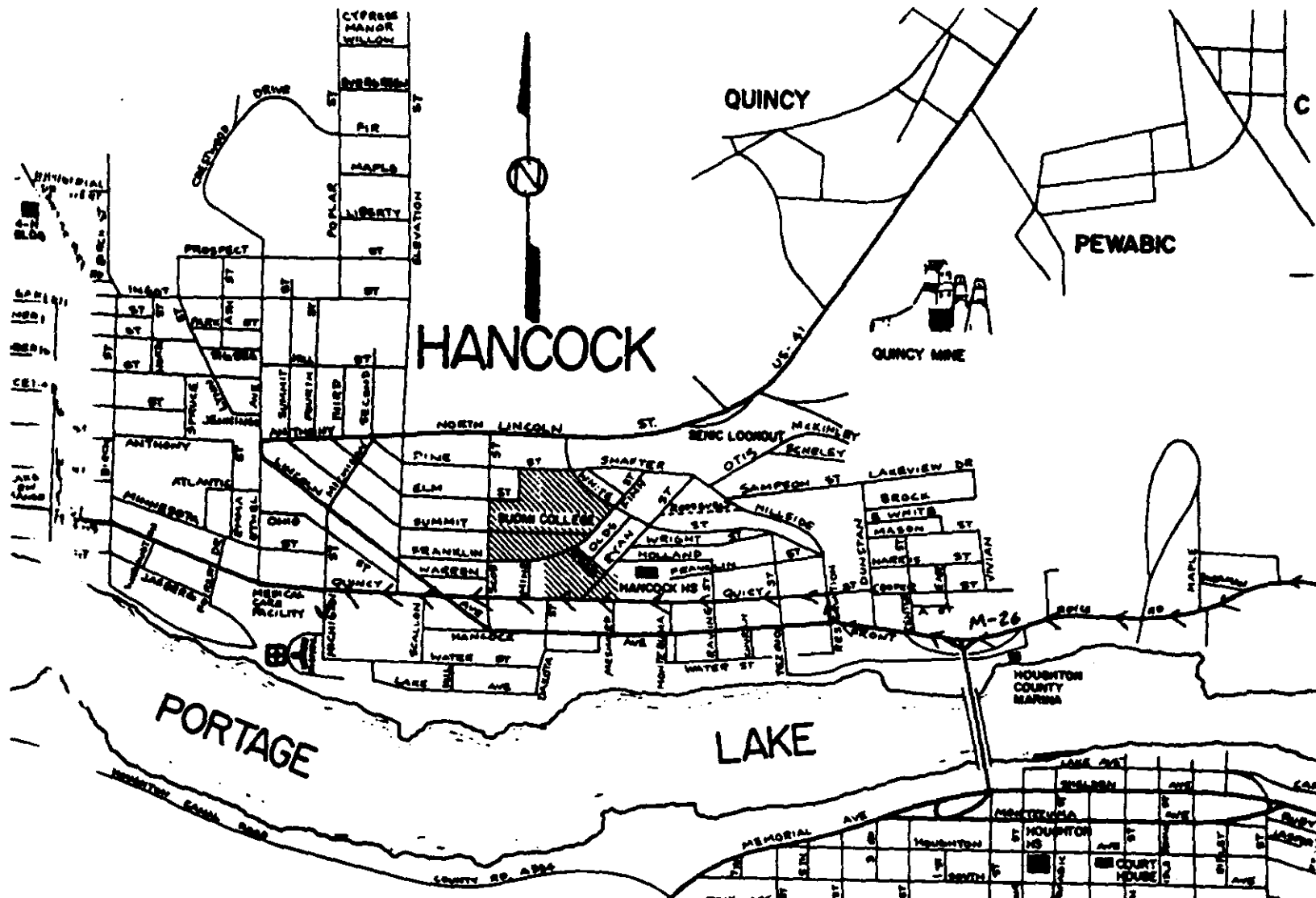
FILE NO:

DRAWN:

CHECKED:

APPROVED:

DRAFTER JPA



GERAGHTY & MILLER, INC.
Environmental Services

HOSPITAL ROUTE MAP

TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN

FIGURE

5

EXHIBIT A
SAMPLING PROCEDURES GUIDANCE

2.4 BULK MATERIALS

Unlike soils which are heterogeneous associations of earthen and manufactured substances, bulk materials are generally a homogeneous collection of a single identifiable product. They are usually contained in bags, drums or hoppers although on occasion large amounts of the material may be piled directly on the ground, either deliberately or as the result of a spill.

Those surfaces exposed to the atmosphere may undergo some chemical alteration or degradation and should be avoided during sample collection. Since the process producing the bulk material may demonstrate some variation with respect to time, it is advisable to collect a series of samples as one composite to represent the material.

Bulk materials in an unconsolidated state may be readily collected by a stainless steel scoop. When the amount of the material is large, a composite can be collected by the use of a grain thief (see Figure 2-6). This device is essentially a long hollow tube with evenly spaced openings along its length. This tube is placed inside an outer sleeve with similar openings and forced into the material. The inner sleeve is rotated until its openings align with those on the outer sleeve, thus allowing the material to enter. The inner sleeve is then further rotated sealing the openings, the device is withdrawn, and the sample recovered.

Grain thieves are available in many materials including brass and various plastics. As with other sampling devices, care should be taken to choose a construction material which will not compromise the desired analytical results.

A more detailed treatment of this subject (Bulk Materials) can be found in The Sampling of Bulk Materials by R. Smith and G. V. James, The Royal Society of Chemistry, London (1981). Although this book does not deal specifically with hazardous waste sampling, the concepts discussed, especially on the subject of the establishment of a sampling scheme, are readily applicable.

inspection program and provide for a corrective action as necessary;

- Collaborate with other laboratories in establishing quality levels, as appropriate; and,
- Ensure that all data are properly recorded and archived.

All laboratory procedures are documented in writing as either Standard Operating Procedures (SOP) or Method Procedures (MP) which are edited and controlled by the quality assurance manager. Internal QC procedures for analytical services will be conducted by the laboratory contracted for the Torch Lake drum removal effort in accordance with their SOPs and the individual method requirements. These specifications include the type of audits required (sample spikes, surrogate spikes, reference samples, controls, blanks), the frequency of each audit, the compounds to be used for sample spikes and surrogate spikes, and the QC acceptance criteria for these audits.

The laboratory will document, in each data package provided, that both initial and ongoing instrument and analytical QC functions have been met. Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient sample volume is available. The sample bottle requirements for the Torch Lake sampling effort, once established, will be designed so that sufficient volume of sample is collected to enable reanalysis, if necessary.

6.0 REFERENCES¹

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¹ Copies of relevant portions of these references will be supplied to the USEPA on request.

APPENDIX B
SAMPLING AND ANALYSIS PLAN

**FINAL SAMPLING AND ANALYSIS PLAN
TORCH LAKE DRUM REMOVAL
HOUGHTON COUNTY, MICHIGAN**

July 1991

Prepared For:

Universal Oil Products Co, Inc.
Quincy Mining Company
Quincy Development Corp.
Houghton County Department of Public Works
Superior Crafts, Inc.
Rudolf Kump

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2.0 SAMPLING OBJECTIVES	2
3.0 EQUIPMENT AND PERSONNEL	3
4.0 SAMPLING PROCEDURES	4
4.1 DRUM OPENING	4
4.2 DRUM SAMPLING	4
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4.2.2 LIQUIDS SAMPLING	5
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5.1 SAMPLE PARAMETERS	12
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EXHIBITS

- Exhibit A - Sampling Procedures Guidance
- Exhibit B - Example Chain-of-Custody Form
- Exhibit C - Regulatory Information on Ignitability and TCLP
- Exhibit D - Target Compound List: VOCs and ABNs

1.0 INTRODUCTION

On behalf of Universal Oil Products Co., Inc., Quincy Mining Company, Quincy Development Corp., Houghton County Department of Public Works, Superior Crafts, Inc., and Rudolf Kump ("Respondents"), Geraghty & Miller, Inc. has developed this Sampling and Analysis Plan (SAAP) for the drum removal effort to take place at the Torch Lake Superfund Site in Houghton County, Michigan. This SAAP is a supplement to the Torch Lake Drum Removal Work Plan ("Work Plan") developed in response to the United States Environmental Protection Agency (USEPA) administrative order by consent (the Order). The sampling techniques outlined in this SAAP were selected to provide protection of human health and the environment during drum characterization and while being consistent with the goals of the NCP, the Order, and appropriate EPA guidance.

A listing of the objectives of the sampling effort, the equipment needed, a brief description of the on-land drum sampling procedures, and the analytical requirements of the drum removal activity are outlined in this report. Health and Safety issues related to the drum removal effort are presented in the site-specific Health and Safety Plan (Appendix A of the Torch Lake Drum Removal Work Plan). Any off-shore sampling conducted will be similar to the on-land procedures described below for bulk solids, and no drum opening procedures or sampling of drums containing liquid will be performed under water. Specific underwater sampling techniques, if the underwater sampling option is selected, will be presented following the selection of a qualified underwater subcontractor. The reader is referred to the Work Plan for details regarding site location, history, and previous drum sampling results.

2.0 SAMPLING OBJECTIVES

The major objective of the drum sampling effort at the Torch Lake Superfund Site is to determine the existence of hazardous substances in the drums. This objective will be accomplished through representative sampling and analysis for the parameters outlined in Section 5.0 of this SAAP. If the drums are determined to contain CERCLA hazardous substances or RCRA hazardous wastes, they will be removed from the site. A secondary objective of the sampling effort is to determine the appropriate disposal or treatment technique for the wastes determined to contain hazardous substances. Although certain disposal facilities will require additional parameters to be run on representative samples of any material prior to disposal, analyses of the data obtained from this sampling effort will be valuable in determining disposal options.

3.0 EQUIPMENT AND PERSONNEL

The equipment and personnel necessary to implement the sampling procedures as outlined in Section 4.0 of this report include:

- Backhoe with Non-Sparking Drum Spike
- Non-Sparking Bung Wrench
- Stainless Steel Scoop or Spoon (Solid Drum Samples)
- Glass Sampling Tube (Liquid Drum Samples)
- Chisel and Mallet (Slag Drum Samples)
- Sample Containers
- Personal Protective Equipment
- Analytical Laboratory
- Backhoe Operator
- Site Safety Officer
- Drums Sampling Personnel (2)

The specific role played by each of the above specialized personnel and equipment is expanded upon in Section 4.0 of this report.

4.0 SAMPLING PROCEDURES

4.1 DRUM OPENING

Based on previous site inspections by the USEPA and Geraghty & Miller, it has been observed that the majority of drums on-site are open and contain solid materials. It is anticipated, however, that a limited number of non-empty drums may be sealed and therefore require opening prior to sampling. If a non-empty drum is intact and sealed but is capped by a loose lid or bung that may be easily removed, removal of the loose bung or lid may be performed by hand or with the use of non-sparking tools (e.g., bung wrench). If the non-empty container appears to be tightly sealed and/or in poor condition, the drum will be opened using a non-sparking drum spike attached to a backhoe, drum grapppler, or similar piece of equipment.

4.2 DRUM SAMPLING

All drums sampled will be assigned a unique drum number and will be photo documented. The actual procedure for the sampling of drums will vary depending on the phase of the drum contents (i.e., liquid or solid). The different procedures applied to the sampling of liquid and solid drum contents are presented below. Also presented in this section is the rationale for the selection of a representative sample of drums containing similar waste materials.

4.2.1 Solids Sampling

The majority of drums on-site contain solid materials. The consistency of the solid types of material observed in the drums differs dramatically, however, and thus sample

retrieval must differ as well. The rock-hard vitrified slag, for example, will require the use of a pick or chisel to dislodge a sample from the monolithic block of material found in the typical slag drum. The semi-solid tar-like material or the soil-like solid material encountered in some of the other drums will more easily be retrieved with a stainless steel spoon, scoop, or trier. The method for sampling the majority of the non-slag solid or semi-solid materials in the drums will closely follow those given in USEPA guidance for the sampling of bulk materials (USEPA 1984) as given in Exhibit A. A summary of the procedure to be used is as follows:

1. Insert the decontaminated sampling device into the waste material.
2. Rotate the sampling device to cut a core of the material.
3. Slowly withdraw the sampling device with the slot or open (concave) end of the device facing upward.
4. Transfer the sample directly into the proper sample container with the aid of a decontaminated stainless steel spoon.

4.2.2 Liquids Sampling

The few drums anticipated to contain liquids on-site will be sampled using a glass tube 6 mm to 16 mm wide (necessary diameter will be dependent upon liquid viscosity). The tube will be lowered into the drum by hand, and the top of the tube will then be sealed with a stopper or thumb prior to being withdrawn. The sample will then be transferred to the appropriate sample container. The specific liquids sampling procedure will closely follow the procedure given in USEPA guidance for the sampling of liquid containerized wastes (USEPA 1984) as given in Exhibit A. A summary of the procedure to be used is as follows:

- 1) Insert a length of new glass tubing slowly into the drum opening to approach the bottom of the container without actually touching the bottom of the container. Attempt to keep a minimum of 30 cm of the tube above the container opening.
- 2) Allow the waste in the drum to reach its natural level within the tube.
- 3) Cap the top of the tube with a stopper or thumb, and carefully remove the tube and insert the uncapped end into a sample container.
- 4) Release the stopper/thumb at the top of the tube and allow the liquids to flow into the sample container, filling the container to approximately 90% of its capacity.
- 5) Repeat steps 1 through 4 with the same tube if more sample volume is needed.
- 6) Break the used glass tube and place it within the drum.

4.2.3 Selection of Representative Samples

Prior inspections of the waste materials contained within the drums have revealed that some of the drums have obviously similar contents. The waste materials observed in the vast majority of the drums on the Torch Lake site have been limited to five different materials. These five materials, listed in order from most common to least common on the site, are as follows:

- Vitrified Slag
- Tar-Like Semi-Solid
- Light Brown Solid
- Black Peat-Like Solid
- Black Liquid

After the potentially hazardous drums have been staged and opened, they will be visually classified into these five categories. One drum sample will then be collected in each category for every five drums found in that category per location. For example, if three drums of light brown solid and seven drums of tar-like semi-solid are removed in Area 1, one sample of the light brown solid and two samples of the tar-like semi-solid will be collected analyzed for the parameters listed in Section 5.0 of this report.

An exception to the representative sampling program will occur on drums previously sampled by the USEPA. None of the drums previously sampled by the USEPA will be resampled under this removal program, and the results obtained from the previous sampling round will be used to characterize the waste within the sample container. Previous drum samples and their analytical results are as follows:

<u>Date</u>	<u>Location</u>	<u>Sample Number</u>	<u>Description</u>	<u>Result</u>
6/21/89	Area 1	S-57	Solid	N.H.
6/21/89	Area 1	S-58	Solid	N.H.
6/21/89	Area 1	S-59	Solid	N.H.
8/01/90	Area 1	S-63	Lt Brn Solid	N.H.
8/01/90	Area 1	S-64	Tar-Like Semi-solid	H.S.
8/01/90	Area 1	S-65	Lt Brn Solid	N.D
8/01/90	Area 1	S-66	Black Solid	H.S.
6/21/89	Area 2	S-61	Slag	N.H.
6/21/89	Area 2	S-62	Oily/Greasy Rags	H.S.
8/01/90	Area 2	S-67	(Same Drum As Above)	H.S.
8/01/90	Area 3	S-70	Slag	N.H.
8/01/90	Area 3	S-71	Slag	N.H.
6/21/89	Area 4	S-66	Liquid	N.H.
6/21/89	Area 4	S-67	Solid	N.H.
6/21/89	Area 4	S-69	Liquid	N.H.
8/01/90	Area 4	S-68	Black Liquid	H.W.
8/01/90	Area 4	S-69	Tar-Like Semi-Solid	H.S.

For the results column of the preceding table, "N.H." signifies that no significant detections of hazardous substances were noted, "H.S." signifies that hazardous substances were detected in the sample, and "H.W." denotes that the sample has been classified as a hazardous waste. Note that in three cases (S-66, S-67, and S-69) the same sample number was assigned to samples from different drums sampled at different times. Note also that in at least one instance, the same drum was sampled at different times and was assigned different sample numbers. Most of the drums previously sampled by USEPA contractors remain clearly marked. In the event that a drum cannot be matched with a sampling result, that drum will no longer be exempt from the representative sampling procedure.

An additional exception to the representative sampling procedure described above will occur with the sampling of the slag drums. Three slag drums have already been characterized on the site, and all three have proven to be non-hazardous (Donahue 1990; Weston 1990). To re-confirm the non-hazardous nature of the slag, two slag drums from Area 2 and two slag drums from Area 3 will be sampled and analyzed for TCLP metals, the only potential contaminants of concern in the slag. No other slag drums will be sampled or analyzed during the drum removal process. Due to the easily-identified appearance of the slag, the chemical homogeneity of the slag material on the Torch Lake Site, and the extremely low and clearly non-hazardous existing TCLP data on the slag, enough data will exist following this sampling round to decisively classify all the slag drums as non-hazardous.

The Respondents reserve the right to increase the number of drums sampled per waste category for the representative sampling protocol, and to resample and reanalyze drums that produce what appear to be false positive analytical results.

4.3 DECONTAMINATION

Each drum will be sampled with new or decontaminated sampling equipment. For the sampling of liquids, a new glass rod will be used on each drum sampled. When sampling solids, all pieces of equipment coming in direct contact with the sample must first undergo the following decontamination procedure:

- 1) Wash in non-phosphate soap and water mixture
- 2) Rinse with potable water
- 3) Rinse with distilled water
- 4) Rinse with isopropyl alcohol
- 5) Allow to air dry
- 6) Wrap decontaminated sampling implement in aluminum foil

All heavy equipment will be steam-cleaned prior to being moved from one drum area to another, or before being transported off-site for any reason. The decon water from sampling operations and equipment clean-up will not be collected, and will be discharged on-site following decontamination procedures.

4.4 SAMPLE DOCUMENTATION, PREPARATION, AND SHIPMENT

The bound field logbook maintained by Geraghty and Miller will provide the means of recording data collection activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

The title page of the logbook will contain the following:

TZCH11201.TORVFINLSAAP.EFT

- project name;
- project start date; and,
- end date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, level of personal protection being used, and the signature of the person making the entry will be entered. The names of all field investigation team personnel, oversight personnel, and site visitors will also be recorded in the field logbook.

All field measurements made and samples collected will be recorded in the logbook. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station shall be recorded. The equipment used to collect samples will be noted, along with the time of sampling, sample description, identification number, and number and types of containers. Field duplicate samples, which will receive an entirely separate identification number, will also be properly identified in the logbook.

If photographs are taken, the photo number, location, orientation, and key features in the photo will be documented in the logbook.

All entries in logbooks will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark and initialed by the person making the correction.

Prior to shipment of the samples to the laboratory, the sample containers will be cleaned, labels will be taped with transparent tape, and sample jars will be placed in a Zip-Loc™ bag or equivalent. The samples will be shipped (via Federal Express) in a cooler

chilled to 4° C with ice.

Samples will be accompanied by a properly completed chain-of-custody form (Exhibit B) enclosed in each sample box or cooler. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

Federal Express will not be required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals will be covered with clear plastic tape. The cooler will be strapped shut with strapping tape in at least two locations.

5.0 ANALYTICAL REQUIREMENTS

5.1 SAMPLE PARAMETERS

All non-slag, potentially hazardous samples that are collected from the drums during this removal effort will be analyzed for:

- Flash Point (Ignitability)
- Toxicity Characteristic Leaching Procedure (TCLP) Metals
- Target Compound List (TCL) Volatile Organic Compounds (VOCs)
- TCL Acid/Base Neutral Semivolatile Organic Compounds (ABNs)

The slag samples collected will be analyzed for TCLP metals only as noted in Section 4.2.3 of this report.

The analysis of these parameters will enable the Respondents to the Order and the OSC to make a decision as to the hazardous content of the materials contained in the drums. The descriptions of the characteristics of ignitability and toxicity are given in 40 CFR 261.20 and 40 CFR 261.24, respectively. The description of the TCLP is given in 40 CFR Part 268 Appendix I. These regulatory references are provided in Exhibit C. The TCL of VOCs and ABNs are provided in Exhibit D.

5.2 QUALITY CONTROL

Field duplicate and matrix spike samples will be analyzed to assess the quality of the data. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and

measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

The general level of the quality control (QC) effort will be one field duplicate for every 10 or fewer investigative samples. One MS/MSD sample will be run for every 20 or fewer investigative samples collected for organic analyses. Non-aqueous MS/MSD samples require no extra volume for VOCs or ABNs.

Internal (laboratory) QC procedures are specified in the 1990 CLP SOWs for organics and inorganics or the method protocols. These specifications include the types of QC checks required (method blanks, reagent/preparation blanks, matrix spike and matrix spike duplicates, calibration standards, internal standards, surrogate standards, the frequency of each audit, the specific calibration check standards, laboratory duplicate analysis), compounds and concentrations to be used, and the QC acceptance criteria for these audits.

Laboratories have a written QA/QC program which provides rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory's QA Department, which is independent of the operating departments, and is directed by the QA Manager (QAM).

The stated objectives of a laboratory QA/QC program are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures;
- Ensure that all analytical procedures are conducted according to sound scientific principles and have been validated;
- Monitor the performance of the laboratory by a systemic

2.4.1 METHOD II-7: SAMPLING OF BULK MATERIAL WITH A SCOOP OR TRIER

Discussion

A typical sampling trier (Figure 2-5) is a long tube with a slot that extends almost its entire length. The tip and edges of the tube slot are sharpened to allow the trier to cut a core of the material to be sampled when rotated after insertion into the material. Sampling triers are usually made of stainless steel with wooden handles. They are about 61 to 100 cm long and 1.27 to 2.54 cm in diameter. They can be purchased readily from laboratory supply houses.

A laboratory scoop or garden variety trowel can also be used to sample bulk material. The trowel looks like a small shovel. The blade is usually about 7 by 13 cm with a sharp tip. A laboratory scoop is similar to the trowel, but the blade is usually more curved and has a closed upper end to permit the containment of material. Scoops come in different sizes and shapes. Stainless steel or polypropylene scoops with 7 by 15 cm blades are preferred. A trowel can be bought from hardware stores; the scoop can be bought from laboratory supply houses.

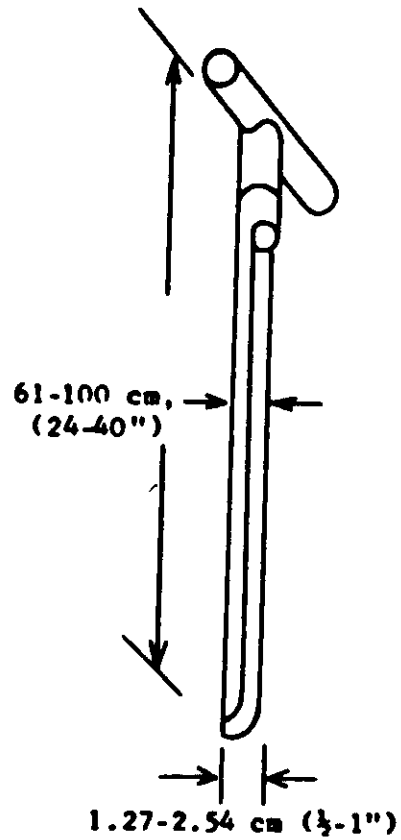
Uses

The use of the trier is similar to that of the grain sampler discussed in Method II-8. It is preferred over the grain sampler when the powdered or granular material to be sampled is moist or sticky.

The trowel or lab scoop can be used in some cases for sampling dry, granular or powdered material in bins or other shallow containers. The lab scoop is a superior choice since it is usually made of materials less subject to corrosion or chemical reactions.

Procedures for Use

1. Insert the precleaned trier into the waste material at a 0 to 45° angle from horizontal. This orientation minimizes the spillage of sample from the sampler. Extraction of samples might require tilting of the containers.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. Transfer the sample into a suitable container with the aid of a spatula and/or brush.
5. If composite sampling is desired, repeat the sampling at different points two or more times and combine the samples in the same sample container.



Source: Reference 4.

Figure 2-5. Sampling trier.

6. Check that a Teflon liner is present in the cap if required. Secure the cap tightly. The chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach supplemented by a minimal holding time. Consult Appendix A for sample containerization and preservation requirements.
7. Label the sample bottle with the appropriate sample tag. Be sure to label the tag carefully and clearly, addressing all the categories or parameters. Complete all chain-of-custody documents and record in the field logbook.
8. Clean and decontaminate sampler after use and between sampling locations as per guidelines presented in Appendix E, Decontamination.

Sources

deVera, E.R., Simmons, B.P., Stephens, R.D., and Storm, D.L. "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA-600/2-80-018. January 1980.

3.3 CONTAINERIZED LIQUIDS

The sampling of tanks, containers, and drums present unique problems not associated with natural water bodies. Containers of this sort are generally closed except for small access ports, manways, or hatches on the larger vessels or taps and bungs on smaller drums. The physical size, shape, construction material, and location of access will limit the types of equipment and methods of collection.

When liquids are contained in sealed vessels, gas vapor pressures build up, sludges settle out, and density layerings develop. The potential for explosive reactions or the release of noxious gases when containers are opened requires considerable safeguards. The vessels should be opened with extreme caution. Preliminary sampling of any headspace gases may be warranted. Section 4.4 details procedures for sampling headspace gases. As a minimum, a preliminary check with an organic vapor analyzer may help determine needed levels of personnel protection and may be of aid in selecting a sampling method.

In most cases it is impossible to observe the contents of these sealed or partially sealed vessels. Since some layering or stratification is likely in any solution left undisturbed over time, a sample must be taken that represents the entire depth of the vessel.

Agitation to disrupt the layers and rehomogenize the sample is physically difficult and almost always undesirable. In vessels greater than 1 meter in depth the method of choice is to slowly, in known increments of length, lower the suction line from a peristaltic pump. Discrete samples can be collected from various depths then combined or analyzed separately. If the depth of the vessel is greater than the lift capacity of the pump, an at-depth water sampler, such as the Kemmerer type discussed in Method III-4, or the ASTM Bomb (Bacon Bomb) may be required. In situations where the reactive nature of the contents are known, a small submersible pump may be used.

When sampling a previously sealed vessel, a check should be made for the presence of a bottom sludge. This is easily accomplished by measuring the depth to apparent bottom then comparing it to the known interior depth. Methods for sampling a bottom sludge are found in Section 2.3.

The sampling of drums for hazardous liquid wastes is a very taxing situation with present equipment. The most widely used method is a glass tube, 6 mm to 16 mm I.D., that is lowered into the drum. The top of the tube is sealed with a stopper or the thumb and the tube withdrawn. The bottom of the tube is then placed over a glass jar, the stopper removed from the top and the contents drained into the containers. After collection of sufficient sample the tube is then broken up into the drum. This method is simple, relatively inexpensive, and quick and collects a sample without having to decontaminate equipment. It does, however, have serious drawbacks. Most low density fluids do not hold well in the glass tubes. A great deal of the potential sample flows out of the bottom of the tube as it is raised from the drum, thereby reducing the representativeness of collected material. Many

variations to this technique have been reported. These include the incorporation of a small suction device (i.e., pipette bulb) to the top of the tube as well as the use of various tube sizes. Some success has been reported with tubes that have been heated at one end then drawn to form a much smaller orifice. This allows the use of larger diameter tubing, therefore a greater volume of sample per attempt, while reducing the material loss from the tube bottom normally associated with larger diameter tubes.

It should be noted that in some instances disposal of the tube by breaking it into the drum may interfere with eventual plans for the removal of its contents. The use of this technique should therefore be cleared with the project officer, or other disposal techniques evaluated.

In many instances a drum containing waste material will have a sludge layer on the bottom (Method III-5). Slow insertion of the sample tube down into this layer and then a gradual withdrawal will allow the sludge to act as a bottom plug to maintain the fluid in the tube. The plug can be gently removed and placed into the sample container by the use of a stainless steel lab spoon. These spoons are relatively inexpensive and can be disposed of in the original waste container with the glass transfer tube.

Designs exist for equipment that will collect a sample from the full depth of a drum and maintain it in the transfer tube until delivery to the sample bottle. These designs include primarily the Composite Liquid Waste Sampler (COLIWASA) and modifications thereof.² The COLIWASA is difficult to properly decontaminate in the field; its applicability is therefore limited to those cases when a sample of the full depth of the drum is absolutely necessary. The COLIWASA can be somewhat modified for this task by making the lift rod of stainless steel, the bottom stopper of Teflon, and the body of glass tubing. In this configuration the glass tube can be broken into the drum leaving only the center rod and the stopper to be decontaminated. In a preliminary investigation where the total number of drums to be sampled is small an equal number of both the center rods and bottom stoppers could be made in advance thus eliminating the time involved for onsite cleanup. Heat shrinkable Teflon tubing or other types of Teflon coating can also be used to cover the stainless steel rod if contact of the stainless steel with the waste is undesirable.

3.3.1 METHOD III-5: COLLECTION OF LIQUID CONTAINERIZED WASTES USING GLASS TUBES

Description

Liquid samples from opened containers (55-gallon drums) are collected using lengths of glass tubing. The glass tubes are normally 122 cm in length and 6 to 16 mm inside diameter. Larger diameter tubes may be used for more viscous fluids if sampling with the small diameter tube is not adequate. The tubing is broken up and discarded in the container after the sample has been collected, eliminating difficult cleanup and disposal problems. This method should not be attempted with less than a two-man sampling team.

Uses

This method provides for a quick, relatively inexpensive means of collecting concentrated containerized wastes. The major disadvantage is from potential sample loss which is especially prevalent when sampling less viscous fluids. Splashing can also be a problem and proper protective clothing (e.g., butyl rubber apron, face shields, boot covers) should always be worn.

Procedures for Use

1. Remove cover from sample container opening.
2. Insert glass tubing slowly to almost the bottom of the container. Try to keep at least 30 cm of tubing above the top of the container.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Cap the top of the tube with a safety-gloved thumb or a rubber stopper.
5. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.
6. Release the thumb or stopper on the tube and allow the sample container to fill to approximately 90 percent of its capacity.
7. Repeat steps 2 through 6 if more volume is needed to fill the sample container.
8. Remove the tube from the sample container and replace the tube in the drum.
9. Cap the sample container tightly with a Teflon-lined cap and affix the sample identification tag.
10. Break the glass sampling tube in such a way that all parts of it are discarded inside the drum. (Note: see the initial discussion to this section for exceptions.)

11. Replace the bung or place plastic over the drum.
12. Place sample container in a Ziplock plastic bag (one per bag).
13. Place each bagged container in a 1-gallon metal paint can (or appropriate sized container) and pack in vermiculite packing material. Place lid on the can.
14. Mark the sample identification number on the outside of each paint can and complete chain-of-custody log and the field logbook.

Optional Method (if sample of bottom sludge is desired)

1. Remove cover from container opening.
2. Insert glass tubing slowly almost to the bottom of the container. Try to keep at least 30 cm of tubing above the top of the container.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Gently push the tube towards the bottom of the drum into the sludge layer. Do not force it.
5. Cap the top of the tube with a safely-gloved thumb or rubber stopper.
6. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.
7. Release the thumb or stopper on the tube and allow the sample container to fill to approximately 90 percent of its capacity. If necessary, the sludge plug in the bottom of the tube can be dislodged with the aid of a stainless steel laboratory spatula.
8. Repeat if more volume is needed to fill sample container and recap the tube.
9. Proceed as in Steps 9 through 14 above.

Note:

1. If a reaction is observed when the glass tube is inserted (violent agitation, smoke, light, heat, etc.) the investigator should leave the area immediately.
2. If the glass tube becomes cloudy or smokey after insertion into the drum, the presence of hydrofluoric acid is indicated and a comparable length of rigid plastic tubing should be used to collect the sample.

3. When a solid is encountered in a drum (either layer or bottom sludge) the optional method described above may be used to collect a core of the material, or the material may be collected with a disposable scoop attached to a length of wooden or plastic rod.
4. If analysis is to be performed onsite, packing steps 12 and 13 may be deleted. These steps are necessary for transporting and/or shipping samples.

Sources

American Society for Testing and Materials. "Standard Recommended Practices for Sampling Industrial Chemicals," ASTM E-300-73.

U.S. Environmental Protection Agency, "Technical Methods for Investigating Sites Containing Hazardous Substances, Technical Monograph 1-29, Draft," Ecology and the Environment, June 1981.

EXHIBIT B

EXAMPLE CHAIN-OF-CUSTODY FORM

Delivery Method: ☐ In Person ☐ Common Carrier ☐ Lab Courier ☐ Other

EXHIBIT C

REGULATORY INFORMATION ON IGNITABILITY AND TCLP

for longer than 90 days due to the regulations under this Part becomes an owner/operator of a storage facility and must obtain a RCRA permit. Such a facility may qualify for interim status upon compliance with the regulations governing interim status under 40 CFR 270.70).

(2) An owner/operator of a hazardous waste treatment, storage, or disposal facility stores such wastes in tanks or containers solely for the purpose of the accumulation of such quantities of hazardous waste as necessary to facilitate proper recovery, treatment, or disposal and:

(i) Each container is clearly marked to identify its contents and the date each period of accumulation begins;

(ii) Each tank is clearly marked with a description of its contents, the quantity of each hazardous waste received, and the date each period of accumulation begins, or such information for each tank is recorded and maintained in the operating record at that facility. Regardless of whether the tank itself is marked, an owner/operator must comply with the operating record requirements specified in § 264.73 or § 265.73.

(3) A transporter stores manifested shipments of such wastes at a transfer facility for 10 days or less.

(b) An owner/operator of a treatment, storage or disposal facility may store such wastes for up to one year unless the Agency can demonstrate that such storage was not solely for the purpose of accumulation of such quantities of hazardous waste as are necessary to facilitate proper recovery, treatment, or disposal.

(c) A owner/operator of a treatment, storage or disposal facility may store such wastes beyond one year; however, the owner/operator bears the burden of proving that such storage was solely for the purpose of accumulation of such quantities of hazardous waste as are necessary to facilitate proper recovery, treatment, or disposal.

(d) If a generator's waste is exempt from a prohibition on the type of land disposal utilized for the waste (for example, because of an approved case-by-case extension under § 268.5, an approved § 268.6 petition, or a national capacity variance under subpart C),

the prohibition in paragraph (a) of this section does not apply during the period of such exemption.

(e) The prohibition in paragraph (a) of this section does not apply to hazardous wastes that meet the treatment standards specified under §§ 268.41, 268.42, and 268.43 or the treatment standards specified under the variance in § 268.44, or, where treatment standards have not been specified, is in compliance with the applicable prohibitions specified in § 268.32 or RCRA section 3004.

(f) Liquid hazardous wastes containing polychlorinated biphenyls (PCBs) at concentrations greater than or equal to 50 ppm must be stored at a facility that meets the requirements of 40 CFR 761.65(b) and must be removed from storage and treated or disposed as required by this part within one year of the date when such wastes are first placed into storage. The provisions of paragraph (c) of this section do not apply to such PCB wastes prohibited under § 268.32 of this part.

[51 FR 40642, Nov. 7, 1986; 52 FR 21017, June 4, 1987, as amended at 52 FR 25791, July 8, 1987; 54 FR 36972, Sept. 6, 1989]

APPENDIX I—TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

NOTE: The TCLP is published in Appendix II of part 261.

[55 FR 11876, Mar. 29, 1990]

EFFECTIVE DATE NOTE: At 55 FR 11876, Mar. 29, 1990, Appendix I of Part 261 was revised, effective September 25, 1990. For the convenience of the user, the superseded text is set forth below:

APPENDIX I—TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual contaminants are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, the TCLP need not be run.

2.0 SUMMARY OF METHOD

(See Figure 1)

2.1 For liquid wastes (i.e., those containing insignificant solid material), the waste, after filtration through a 0.6- to 0.8-um glass fiber filter, is defined as the TCLP extract.

2.2 For wastes comprised of solids or for wastes containing significant amounts of solid material, the particle-size of the waste is reduced (if necessary), the liquid phase, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatiles (See Table 1). Following extraction, the liquid extract is separated from the solid phase by 0.6- to 0.8-um glass fiber filter filtration.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these liquids are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 *Agitation apparatus:* An acceptable agitation apparatus is one which is capable of rotating the extraction vessel in an end-over-end fashion (See Figure 2) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessel:

4.2.1 *Zero-Headspace Extraction Vessel (ZHE):* This device is for use only when the waste is being tested for the mobility of volatile constituents (see Table 1). The ZHE is an extraction vessel that allows for liquid/solid separation within the device, and which effectively precludes headspace (as depicted in Figure 3). This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without having to open the vessel (see Step 4.3.1). These vessels shall have an internal volume of 500 to 600 mL and be equipped to accommodate a 90-mm filter. Suitable ZHE devices known to EPA are identified in Table 3. These devices contain viton O-rings which should be replaced frequently.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If

it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

4.2.2 When the waste is being evaluated for other than volatile contaminants, an extraction vessel that does not preclude headspace (e.g., a 2-liter bottle) is used. Suitable extraction vessels include bottles made from various materials, depending on the contaminants to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used over other types of glass, especially when inorganics are of concern. Plastic bottles may be used only if inorganics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.2.3 Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 9.0) refers to pounds-per-square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

4.3 *Filtration Devices:* It is recommended that all filtrations be performed in a hood.

4.3.1 *Zero-Headspace Extraction Vessel* (see Figure 3): When the waste is being evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter, and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an is-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 *Filter Holder:* When the waste is being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is used. Suitable filter holders range

from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Step 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (Filter holders having an internal capacity of 1.5 L or greater and equipped to accommodate a 142 mm diameter filter are recommended). Vacuum filtration is only recommended for wastes with low solids content (<10%) and for highly granular (liquid-containing) wastes. All other types of wastes should be filtered using positive pressure filtration. Filter holders known to EPA to be suitable for use are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are constituents of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6- to 0.8- μ m, or equivalent. Filters known to EPA to meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1.0 N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1-L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH meters: Any of the commonly available pH meters are acceptable.

4.6 ZHE extract collection devices: TEDLAR[®] bags or glass, stainless steel or PTFE gas tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions.

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the TEDLAR[®] bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended in these cases.

4.6.2 If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR[®] bag may be used for both the ini-

tial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR[®] bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE extraction fluid transfer devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a constant displacement pump, a gas tight syringe, pressure filtration unit (See Step 4.3.2), or another ZHE device).

4.8 Laboratory balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at or above the method detection limit of the analyte(s) of interest. For non-volatile extractions, ASTM Type II water, or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.1.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.1.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.1.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at $90 \pm 5^\circ\text{C}$, bubble a contaminant-free inert gas (e.g., nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.2 1.0 N Hydrochloric acid (HCl) made from ACS reagent grade.

5.3 1.0 N Nitric acid (HNO_3) made from ACS reagent grade.

5.4 1.0 N Sodium hydroxide (NaOH) made from ACS reagent grade.

5.5 Glacial acetic acid (HOAc) ACS reagent grade.

5.6 Extraction fluid:

5.6.1 Extraction fluid #1: This fluid is made by adding 5.7 mL glacial HOAc to 500 mL of the appropriate water (see Step 5.1),

adding 64.3 mL of 1.0 N HCl, and diluting to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 .

5.6.2 Extraction fluid #2: This fluid is made by diluting 5.7 mL glacial HOAc with ASTM Type II water (see Step 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

NOTE: It is suggested that these extraction fluids be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately.

5.7 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 At least two separate representative samples of a waste should be collected. If volatile organics are of concern, a third sample should be collected. The first sample is used in several preliminary TCLP evaluations (e.g., to determine the percent solids of the waste; to determine if the waste contains insignificant solids (i.e., the waste is its own extract after filtration); to determine if the solid portion of the waste requires particle-size reduction; and to determine which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste). These preliminary evaluations are identified in Section 7.0. The second and, if required, third samples are extracted using the TCLP non-volatile procedure (Section 8.0) and volatile procedure (Section 9.0), respectively.

6.3 Preservatives shall not be added to samples.

6.4 Samples can be refrigerated unless refrigeration results in irreversible physical change to the waste (e.g., precipitation).

6.5 When the waste is to be evaluated for volatile contaminants, care should be taken to minimize the loss of volatiles. Samples shall be taken and stored in a manner to prevent the loss of volatile contaminants. If possible, it is recommended that any necessary particle-size reduction should be conducted as the sample is being taken (See Step 8.5).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. If they need to be stored, even for a short period of time, storage shall be at 4°C , and samples for volatiles analysis shall not be allowed to come into contact with the atmosphere (i.e., no headspace). See Section 10.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PRELIMINARY TCLP EVALUATIONS

The preliminary TCLP evaluations are performed on a minimum 100 gram representative sample of waste that will not actually undergo TCLP extraction (designated as the first sample in Step 6.2). These evaluations include preliminary determination of the percent solids of the waste; determination of whether the waste contains insignificant solids, and is therefore, its own extract after filtration; determination of whether the solid portion of the waste requires particle-size reduction; and determination of which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste.

7.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Step 7.4.

7.1.2 If the sample is liquid or multiphase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.1.3 through 7.1.9.

7.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.5 Weigh out a representative subsample of the waste (100 gram minimum) and record the weight.

7.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10-psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2-minute period), filtration is stopped.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.9)}}{\text{Total weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

7.2 *Determination of whether waste is liquid or has insignificant amounts of solid material:* If the sample obviously has a significant amount of solid material, the solid phase must be subjected to extraction; proceed to Step 7.3 to determine if the waste requires particle-size reduction (and to reduce particle-size, if necessary). Determine whether the waste is liquid or has insignificant amounts of solid material (which need not undergo extraction) as follows:

7.2.1 Remove the solid phase and filter from the filtration apparatus.

$$\text{Percent dry solids} = \frac{\text{Weight of dry waste and filter - tared weight of filter}}{\text{Initial weight of waste (Step 7.1.5 or 7.1.1)}} \times 100$$

7.2.4 If the percent dry solids is less than 0.5%, consult Step 8.2 and proceed to Section 8.0 if non-volatiles in the waste are of concern, and to Section 9.0 if volatiles are of interest. In this case, the waste, after filtration is defined as the TCLP extract. If the percent dry solids is greater than or equal to 0.5%, and if the non-volatile TCLP is to be performed, return to the beginning of this Section (7.0) with a new representative

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. But even after applying vacuum or pressure filtration, as outlined in Step 7.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. The original filter is not to be replaced with a fresh filter under any circumstances. Only one filter is used.

7.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 7.1.3) from the total weight of the filtrate-filled container. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.1.5 or 7.1.7. Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

7.2.2 Dry the filter and solid phase at 100±20° C until two successive weighings yield the same value within ±1%. Record final weight.

NOTE: Caution should be taken to insure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or appropriate device.

7.2.3 Calculate the percent dry solids as follows:

waste sample, so that it can be determined if particle-size reduction is necessary (Step 7.3), and so that the appropriate extraction fluid may be determined (Step 7.4) on a fresh portion of the solid phase of the waste. If only the volatile TCLP is to be performed, see the Note in Step 7.4.

7.3 *Determination of whether the wastes requires particle-size reduction (particle-size is reduced during this Step):* Using the solid

portion of the waste, evaluate the solid for particle-size. If the solid has a surface area per gram of material equal to or greater than 3.1 cm², or is smaller than 1 cm in its narrowest dimension (e.g., is capable of passing through a 9.5-mm (0.375-inch) standard sieve), particle-size reduction is not required (proceed to Step 7.4). If the surface area is smaller or the particle-size larger than described above, the solid portion of the waste is prepared for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described above.

NOTE: Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not required; nor is it recommended.

7.4 *Determination of appropriate extraction fluid:* If the solid content is greater than or equal to 0.5% of the waste and if TCLP extraction for non-volatile constituents will take place (Section 8.0), determination of the appropriate fluid (Step 5.6) to use for the non-volatiles extraction is performed as follows.

NOTE: TCLP extraction for volatile constituents entails using only extraction fluid #1 (Step 5.6.1). Therefore, if TCLP extraction for non-volatiles extraction is not required, proceed to section 9.0.

7.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle-size of approximately 1mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500-mL beaker or Erlenmeyer flask.

7.4.2 Add 96.5 mL of reagent water (ASTM Type II) to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, extraction fluid #1 is used. Proceed to Section 8.0.

7.4.3 If the pH from Step 7.4.2 is >5.0, add 3.5 mL 1.0 N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 8.0.

7.5 The sample of waste used for performance of this Section shall not be used any further. Other samples of the waste (see Step 8.2) should be employed for the Section 8.0 and 9.0 extractions.

8.0 PROCEDURE WHEN VOLATILES ARE NOT INVOLVED

Although a minimum sample size of 100 grams (solid and liquid phases) is required, a larger sample size may be more appropriate, depending on the solids content of the waste sample (percent solids, see Step 7.1), whether the initial liquid phase of the waste

will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by the performance of a single TCLP extraction will not be sufficient to perform all of the analyses to be conducted, it is recommended that more than one extraction be performed and that the extracts from each extraction be combined and then aliquoted for analysis.

8.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Step 7.1), weigh out a representative subsample of the waste (100 gram minimum) and proceed to Step 8.9.

8.2 If the sample is liquid or multiphase, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 8.3 to 8.8.

8.3 Pre-weigh the container that will receive the filtrate.

8.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (See Step 4.4).

NOTE: Acid washed filters may be used for all non-volatile extractions even when metals are not of concern.

8.5 Weigh out a representative subsample of the waste (100 gram minimum) and record the weight. If the waste was shown to contain <0.5% dry solids (Step 7.2), the waste, after filtration is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Steps 7.1 or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated after filtration to support the analyses to be performed on the TCLP extract.

8.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

8.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Step 4.3.2). If filtration of the waste at 4° C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample

to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 8.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2-minute period), filtration is stopped.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

8.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see Step 8.13) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a

liquid. But even after applying vacuum or pressure filtration, as outlined in Step 8.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. The original filter is not to be replaced with a fresh filter under any circumstances. Only one filter is used.

8.9 If the waste contains <0.5% dry solids (see Step 7.2), proceed to Step 8.13. If the waste contains >0.5% dry solids (see Step 7.1 or 7.2), and if particle-size reduction of the solid was needed in Step 7.3, proceed to Step 8.10. If particle-size reduction was not required in Step 7.3, quantitatively transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase. Proceed to Step 8.11.

8.10 The solid portion of the waste is prepared for extraction by crushing, cutting, or grinding the waste to a surface area of particle-size as described in Step 7.3. When the surface area of particle-size has been appropriately altered, quantitatively transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste through a sieve that is not Teflon coated should not be done due to avoid possible contamination of the sample. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

8.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \% \text{ solids (Step 7.1)} \times \text{weight of waste filtered (Step 8.5 or 8.7)}}{300}$$

300

Slowly add this amount of appropriate extraction fluid (see Step 7.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction is to take place) shall be maintained at 22 ± 3 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes,

30 minutes, and 1 hour) and vented into a hood.

8.12 Following the 18 ± 2 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 8.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

8.13 The TCLP extract is now prepared as follows:

8.13.1 If the waste contained no initial liquid phase, the filtered liquid material ob-

tained from Step 8.12 is defined as the TCLP extract. Proceed to Step 8.14.

8.13.2 If compatible (e.g., multiple phases will not result on combination), the filtered liquid resulting from Step 8.12 is combined with the initial liquid phase of the waste as obtained in Step 8.7. This combined liquid is defined as the TCLP extract. Proceed to Step 8.14.

8.13.3 If the initial liquid phase of the waste, as obtained from Step 8.7, is not or may not be compatible with the filtered liquid resulting from Step 8.12, these liquids are not combined. These liquids, collectively defined as the TCLP extract, are analyzed separately, and the results are combined mathematically. Proceed to Step 8.14.

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).

C_1 = The concentration of the contaminant of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the contaminant of concern in the second phase (mg/L).

8.15 The contaminant concentrations in the TCLP extract are compared with the thresholds identified in the appropriate regulations. Refer to Section 10.0 for quality assurance requirements.

9.0 PROCEDURE WHEN VOLATILES ARE INVOLVED

The ZHE device is used to obtain TCLP extracts for volatile analysis only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500-mL internal capacity. Although a minimum sample size of 100 grams was required in the Section 8.0 procedure, the ZHE can only accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no liquid (additional) may be forced out by an applied pressure of 80 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

The ZHE is charged with sample only once and the device is not opened until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted. The initial filtrate should be weighed and then stored at 4 °C until either analyzed or recombined with the final extract of the solid.

8.14 Following collection of the TCLP extract, it is recommended that the pH of the extract be recorded. The extract should be immediately aliquoted for analysis and properly preserved (metals aliquots must be acidified with nitric acid to pH <2; all other aliquots must be stored under refrigeration (4 °C) until analyzed). The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals, other than mercury, shall be acid digested. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

Although the following procedure allows for particle-size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible (e.g., particle-size may be reduced easily by crumbling), particle-size reduction (See Step 9.2) should be conducted on the sample as it is being taken. If necessary, particle-size reduction may be conducted during the procedure.

In carrying out the following steps, do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

9.1 Pre-weigh the (evacuated) container which will receive the filtrate (See Step 8.6), and set aside. If using a TEDLAR® bag, all liquid must be expressed from the device, whether it be for the initial or final liquid/solid separation, and an aliquot taken from the liquid in the bag, for analysis. The containers listed in Step 4.6 are recommended for use under the following conditions.

9.1.1 If a waste contains an aqueous liquid phase or if the waste does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the TEDLAR® bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended in these cases.

9.1.2 If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final ex-

tract filtration. However, analysts should use one or the other, not both.

9.1.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR[®] bag or the syringe may be used. If the syringe is used, discard the first 5 mL liquid expressed from the device. The remaining aliquots are used for analysis.

9.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to molaten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 9.0, Step 7.1 and/or 7.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

9.3 If the waste is 100% solid (see Step 7.1), weigh out a representative subsample (25 gram maximum) of the waste, record weight, and proceed to Step 9.5.

9.4 If the waste was shown to contain <0.5% dry solids (Step 7.2), the waste, after filtration is defined as the TCLP extract. Enough of the sample should be filtered so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >0.5% dry solids (Steps 7.1 and/or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size to charge into the ZHE. The appropriate sample size recommended is as follows:

9.4.1 For wastes containing >5% solids (see Step 7.1), weigh out a representative 500 gram sample or waste and record the weight.

9.4.2 For wastes containing >5% solids (see Step 7.1), the amount of waste to charge into the ZHE is determined as follows:

Weight of waste to charge ZHE	25	% solids (Step 7.1)	100
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Weigh out a representative subsample of the waste of the appropriate size and record the weight.

9.5 If particle-size reduction of the solid portion of the waste was required in Step 7.3, proceed to Step 9.6. If particle-size reduction was not required in Step 7.3, proceed to Step 9.7.

9.6 The waste is prepared for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle-size as described in Step 7.3. Wastes

and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle-size reduction. The means used to effect particle-size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle-size has been appropriately altered, proceed to Step 9.7.

9.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration.

9.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extraction collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 9.4, to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace (into a hood) slowly out of the ZHE device. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Step 7.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Step 9.12.

9.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force

the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within any 2-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

9.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

$$\text{Weight of extraction fluid} = \frac{20 \times \% \text{ solids (Step 7.1)} \times \text{weight of waste filtered (Step 9.4 or 9.8)}}{100}$$

9.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (see Step 5.6).

9.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

9.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Physically rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Put 5-10 psi behind the piston (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. But even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Step 7.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Step 9.15.

9.11 The liquid phase may now be either analyzed immediately (see Steps 9.13 through 9.15) or stored at 4 °C under minimal headspace conditions until time of analysis. The weight of extraction fluid #1 to add to the ZHE is determined as follows:

ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

9.12.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate the ZHE at 30±2 rpm for 18±2 hours. Ambient temperature (i.e., temperature of room in which extraction is to occur) shall be maintained at 22±3 °C during agitation.

9.13 Following the 18±2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Step 4.2.1, and redo the extraction with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR[®] bag) holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Step 9.9. All extract shall be filtered and collected in the TEDLAR[®] bag is used. If the extract is mil-

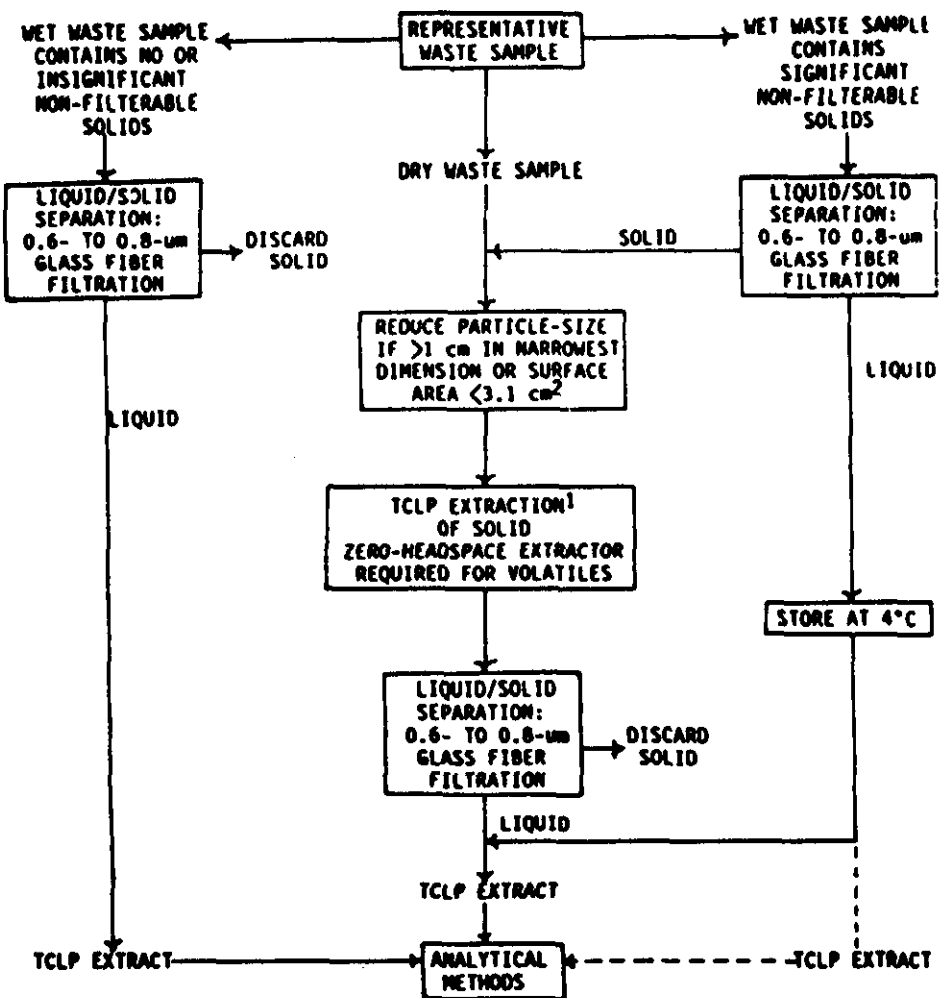


FIGURE 1: TCLP FLOWCHART

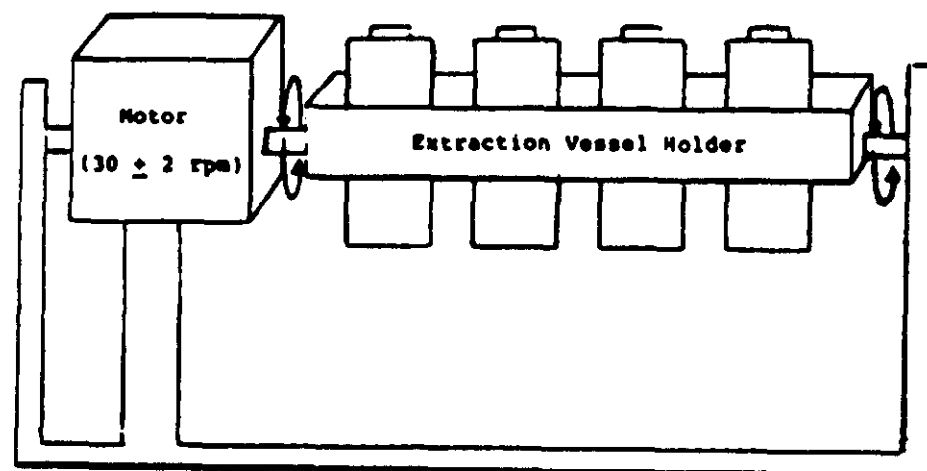


Figure 2: Rotary Agitation

Liquid Inlet/Outlet Valve

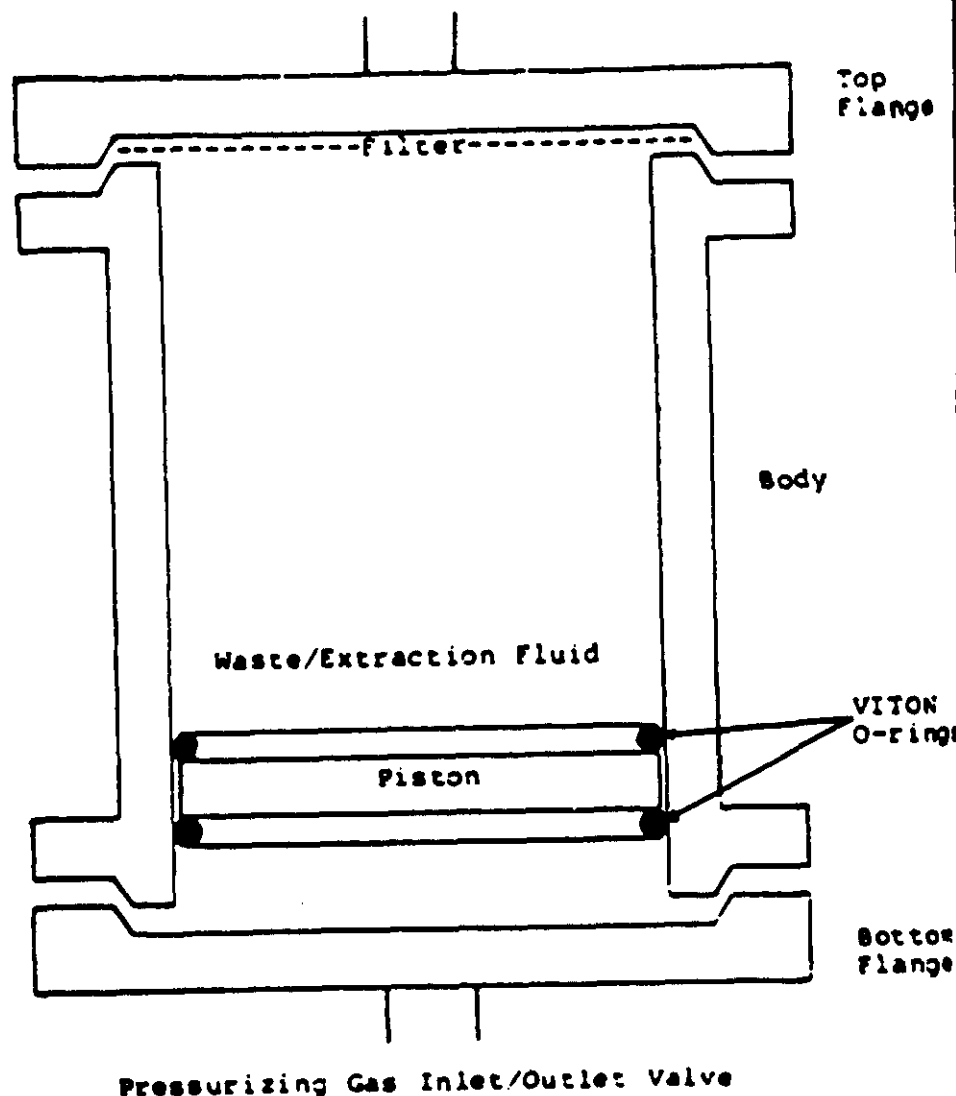


Figure 3: Zero-Headspace Extraction Vessel

(51 FR 40643, Nov. 7, 1986; 52 FR 21018, June 4, 1987)

APPENDIX II—TREATMENT STANDARDS (AS CONCENTRATIONS IN THE TREATMENT RESIDUAL EXTRACT)

(Note: The technologies shown are the basis of the treatment standards. They are not required to be used in meeting the treatment standards.)

Constituents of F001-F005 Spent Solvent Wastes	Waste Treatability Groups For F001-F005 Spent Solvent Wastes (mg/l)			
	Wastewater	Technology Basis ¹	Wastewater Generated by Pharmaceutical Plant ²	All Other ³
Acetone	0.05	SS		0.50
n-Butyl Alcohol	5.00	SS		5.00
Carbon disulfide	1.05	SS		8.81
Carbon tetrachloride	0.05	B		0.88
Chlorobenzene	0.15	SAAC		0.05
Creosote (creylic acid)	2.82	AC		0.75
Cyclohexanone	0.125	SS		0.75
1,2-Dichlorobenzene	0.65	SAAC		0.125
Ethyl acetate	0.05	SS		0.75
Ethylbenzene	0.05	B		0.053
Ethyl ether	0.05	SS		0.75
Isobutanol	5.00	SS		5.00
Methanol	0.25	SS		0.75
Methylene chloride	0.20	B	12.7	0.88
Methyl ethyl ketone	0.05	SS		0.75
Methyl isobutyl ketone	0.05	SS		0.33
Nitrobenzene	0.88	SAAC		0.125
Pyridine	1.12	SAAC		0.33
Tetrachloroethylene	0.079	B		0.05
Toluene	1.12	SAAC		0.33
1,1,1-Trichloroethane	1.05	SS		0.41
1,1,2-Trichloro-1,2,2-trifluoroethane	1.05	SS		0.88
Trichloroethylene	0.082	SAAC		0.081
Trichlorofluoromethane	0.05	B		0.88
Xylene	0.05	AC		0.15

¹ In some instances other technologies achieved somewhat lower treatment values but waste characterization data were insufficient to identify separate treatability groups. Refer to the BDAT background document for a detailed explanation of the determination of the treatment standards.

SS = steam stripping

B = biological treatment

AC = activated carbon

² Wastewaters generated by pharmaceutical plants must be treated to the standards given for all other wastewaters except in the case of methylene chloride.

³ The treatment standards in this treatability group are based on incineration.

(51 FR 40653, Nov. 7, 1986)

APPENDIX III—LIST OF HALOGENATED ORGANIC COMPOUNDS REGULATED UNDER § 268.32

In determining the concentration of HOCs in a hazardous waste for purposes of the § 268.32 land disposal prohibition, EPA has defined the HOCs that must be included in the calculation as any compounds having a carbon-halogen bond which are listed in this Appendix (see § 268.3). Appendix III to Part 268 consists of the following compounds:

Volatiles

Bromodichloromethane
Bromomethane
Carbon Tetrachloride
Chlorobenzene
2-Chloro-1,3-butadiene

Chlorodibromomethane
Chloroethane
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
3-Chloropropene
1,2-Dibromo-3-chloropropane
1,2-Dibromomethane
Dibromomethane
Trans-1,4-Dichloro-2-butene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethylene
Trans-1,2-Dichloroethene
1,2-Dichloropropane
Trans-1,3-Dichloropropene
cis-1,3-Dichloropropene
Iodomethane
Methylene chloride
1,1,1,2-Tetrachloroethane
1,1,2,3-Tetrachloroethane
Tetrachloroethene

(1) It exhibits any of the characteristics of hazardous waste identified in Subpart C.

(2) It has been found to be fatal to humans in low doses or, in the absence of data on human toxicity, it has been shown in studies to have an oral LD 50 toxicity (rat) of less than 50 milligrams per kilogram, an inhalation LC 50 toxicity (rat) of less than 2 milligrams per liter, or a dermal LD 50 toxicity (rabbit) of less than 200 milligrams per kilogram or is otherwise capable of causing or significantly contributing to an increase in serious irreversible, or incapacitating reversible, illness. (Waste listed in accordance with these criteria will be designated Acute Hazardous Waste.)

(3) It contains any of the toxic constituents listed in Appendix VIII and, after considering the following factors, the Administrator concludes that the waste is capable of posing a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported or disposed of, or otherwise managed:

(i) The nature of the toxicity presented by the constituent.

(ii) The concentration of the constituent in the waste.

(iii) The potential of the constituent or any toxic degradation product of the constituent to migrate from the waste into the environment under the types of improper management considered in paragraph (a)(3)(vii) of this section.

(iv) The persistence of the constituent or any toxic degradation product of the constituent.

(v) The potential for the constituent or any toxic degradation product of the constituent to degrade into non-harmful constituents and the rate of degradation.

(vi) The degree to which the constituent or any degradation product of the constituent bioaccumulates in ecosystems.

(vii) The plausible types of improper management to which the waste could be subjected.

(viii) The quantities of the waste generated at individual generation sites or on a regional or national basis.

(ix) The nature and severity of the human health and environmental damage that has occurred as a result of the improper management of wastes containing the constituent.

(x) Action taken by other governmental agencies or regulatory programs based on the health or environmental hazard posed by the waste or waste constituent.

(xi) Such other factors as may be appropriate.

Substances will be listed on Appendix VIII only if they have been shown in scientific studies to have toxic, carcinogenic, mutagenic or teratogenic effects on humans or other life forms.

(Wastes listed in accordance with these criteria will be designated Toxic wastes.)

(b) The Administrator may list classes or types of solid waste as hazardous waste if he has reason to believe that individual wastes, within the class or type of waste, typically or frequently are hazardous under the definition of hazardous waste found in section 1004(5) of the Act.

(c) The Administrator will use the criteria for listing specified in this section to establish the exclusion limits referred to in § 261.5(c).

[45 FR 33119, May 19, 1980, as amended at 55 FR 18726, May 4, 1990]

Subpart C—Characteristics of Hazardous Waste

§ 261.20 General.

(a) A solid waste, as defined in § 261.2, which is not excluded from regulation as a hazardous waste under § 261.4(b), is a hazardous waste if it exhibits any of the characteristics identified in this subpart.

[Comment: § 261.11 of this chapter sets forth the generator's responsibility to determine whether his waste exhibits one or more of the characteristics identified in this subpart.]

(b) A hazardous waste which is identified by a characteristic in this subpart is assigned every EPA Hazardous Waste Number that is applicable as set forth in this subpart. This number must be in complying with the notification requirements of section 3010 of

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the Act and all applicable recordkeeping and reporting requirements under parts 262 through 265, 268, and 270 of this chapter.

(c) For purposes of this subpart, the Administrator will consider a sample obtained using any of the applicable sampling methods specified in Appendix I to be a representative sample within the meaning of Part 260 of this chapter.

[Comment: Since the Appendix I sampling methods are not being formally adopted by the Administrator, a person who desires to employ an alternative sampling method is not required to demonstrate the equivalency of his method under the procedures set forth in §§ 260.20 and 260.21.]

[45 FR 33119, May 1980, as amended at 51 FR 40436, Nov. 7, 1986; 55 FR 22684, June 1, 1990]

§ 261.21 Characteristic of ignitability.

(a) A solid waste exhibits the characteristic of ignitability if a representative sample of the waste has any of the following properties:

(1) It is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has flash point less than 60°C (140°F), as determined by a Pensky-Martens Closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80 (incorporated by reference, see § 260.11), or a Setaflash Closed Cup Tester, using the test method specified in ASTM Standard D-3278-78 (incorporated by reference, see § 260.11), or as determined by an equivalent test method approved by the Administrator under procedures set forth in §§ 260.20 and 260.21.

(2) It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.

(3) It is an ignitable compressed gas as defined in 49 CFR 173.300 and as determined by the test methods described in that regulation or equivalent test methods approved by the Administrator under §§ 260.20 and 260.21.

(4) It is an oxidizer as defined in 49 CFR 173.151.

(b) A solid waste that exhibits the characteristic of ignitability has the EPA Hazardous Waste Number of D001.

[45 FR 33119, May 19, 1980, as amended at 46 FR 35247, July 7, 1981; 55 FR 22684, June 1, 1990]

§ 261.22 Characteristic of corrosivity.

(a) A solid waste exhibits the characteristic of corrosivity if a representative sample of the waste has either of the following properties:

(1) It is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5, as determined by a pH meter using either an EPA test method or an equivalent test method approved by the Administrator under the procedures set forth in §§ 260.20 and 260.21. The EPA test method for pH is specified as Method 5.2 in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (incorporated by reference, see § 260.11).

(2) It is a liquid and corrodes steel (SAE 1020) at a rate greater than 6.35 mm (0.250 inch) per year at a test temperature of 55°C (130°F) as determined by the test method specified in NACE (National Association of Corrosion Engineers) Standard TM-01-69 as standardized in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods" (incorporated by reference, see § 260.11) or an equivalent test method approved by the Administrator under the procedures set forth in §§ 260.20 and 260.21.

(b) A solid waste that exhibits the characteristic of corrosivity has the EPA Hazardous Waste Number of D002.

[45 FR 33119, May 19, 1980, as amended at 46 FR 35247, July 7, 1981; 55 FR 22684, June 1, 1990]

§ 261.23 Characteristic of reactivity.

(a) A solid waste exhibits the characteristic of reactivity if a representative sample of the waste has any of the following properties:

(1) It is normally unstable and readily undergoes violent change without detonating.

(2) It reacts violently with water.

tiphasic, or if the waste contained an initial liquid phase (see Steps 4.6 and 9.1).

Note: An in-line glass fiber filter may be used to filter the material within the ZHE when it is suspected that the glass fiber filter has been ruptured.

9.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Step 9.13 is defined as the TCLP extract. If the waste contained in initial liquid phase, the filtered liquid material obtained from Step 9.13 and the initial liquid phase (Step 9.9) are collectively defined as the TCLP extract.

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).

C_1 = The concentration of the contaminant of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the contaminant of concern in the second phase (mg/L).

9.16 The contaminant concentrations in the TCLP extract are compared with the thresholds identified in the appropriate regulations. Refer to Section 10.0 for quality assurance requirements.

10.0 QUALITY ASSURANCE REQUIREMENTS

10.1 All data, including quality assurance data, should be maintained and available for reference or inspection.

10.2 A minimum of one blank (extraction fluid #1) for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring.

10.3 For each analytical batch (up to twenty samples), it is recommended that a matrix spike be performed. Addition of matrix spikes should occur once the TCLP extract has been generated (i.e., should not occur prior to performance of the TCLP procedure). The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract and for determining if matrix interferences exist in analyte detection.

10.4 All quality control measures described in the appropriate analytical methods shall be followed.

10.5 The method of standard addition shall be employed for each analyte if: 1) recovery of the compound from the TCLP extract is not between 50 and 150%, or 2) if the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more

9.15 Following collection of the TCLP extract, the extract should be immediately aliquoted for analysis and stored with minimal headspace at 4 °C until analyzed. The TCLP extract will be prepared and analyzed according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

than one extraction is being run on samples of the same waste (up to twenty samples), the method of standard addition need be applied only once and the percent recoveries applied to the remainder of the extractions.

10.6 Samples must undergo TCLP extraction within the following time period after sample receipt: Volatiles, 14 days; Semi-Volatiles, 40 days; Mercury, 28 days; and other Metals, 180 days. Extraction of the solid portion of the waste should be initiated as soon as possible following initial solid/liquid separation. TCLP extracts shall be analyzed after generation and preservation within the following periods: Volatiles, 14 days; Semi-Volatiles, 40 days; Mercury, 28 days; and other Metals, 180 days.

TABLE 1—VOLATILE CONTAMINANTS¹

Compound	CAS No.
Acetone	67-64-1
n-Butyl alcohol	71-36-8
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Methylene chloride	75-08-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	106-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
Trichloroethylene	78-01-6
Trichlorofluoromethane	75-68-4
Xylene	1330-20-7

¹ Includes compounds identified in the Land Disposal Restrictions Rule. If any or all of these compounds are of concern, the zero-headspace extractor vessel shall be used. If other (non-volatile) compounds are of concern, the conventional bottle extractor shall be used.

TABLE 2—SUITABLE ROTARY AGITATION APPARATUS¹

Company	Location	Model
Associated Design and Manufacturing Company	Alexandria, VA (703) 548-5888	4-vessel device, 6-vessel device
Lars Labs Manufacturing	Whitmore Lake, MI (313) 448-4118	10-vessel device, 5-vessel device
MA Machine Shop and Laboratory	Santauro, PR (809) 752-4004	16-vessel device
EPRI Extractor		6-vessel device ²

TABLE 2—SUITABLE ROTARY AGITATION APPARATUS¹—Continued

Company	Location	Model
REXNORD	Milwaukee, WI (414) 643-2860	6-vessel device
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel device

¹ Any device that rotates the extraction vessel in an end-over-end fashion at 30 \pm 2 rpm is acceptable.

² Although this device is suitable, it is not commercially made. It may also require retrofitting to accommodate ZHE devices.

TABLE 3—SUITABLE ZERO-HEADSPACE EXTRACTOR VESSELS

Company	Location	Model No.
Associated Design & Manufacturing Co.	Alexandria, VA, (703) 548-5888	3740-ZH8, Gas Pressure Device.
Millipore Corp.	Bedford, MA, (800) 225-3384	SD1 P581 C5, Gas Pressure Device.
Analytical Testing & Consulting Services, Inc.	Warrington, PA, (215) 343-4490	C202, Mechanical Pressure Device.

TABLE 4—SUITABLE FILTER HOLDERS¹

Company	Location	Model	Size
Nuclepore Corp.	Pleasanton, CA, (800) 882-7711	425810	142 mm.
		410400	47 mm.
Micro Filtration Systems	Dublin, CA, (415) 828-8010	302400	142 mm.
Millipore Corp.	Bedford, MA, (800) 225-3384	YT30142YW	142 mm.
		XX1004700	47 mm.

¹ Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern. The 142 mm size filter holder is recommended.

TABLE 5—SUITABLE FILTER MEDIA

Company	Location	Model	Pore Size ¹
Whatman Laboratory Products, Inc.	Cifton, NJ, (201) 773-5800	QFF	0.7

¹ Nominal pore size.

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(3) It forms potentially explosive mixtures with water.

(4) When mixed with water, it generates toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(5) It is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment.

(6) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

(7) It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

(8) It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53 or a Class B explosive as defined in 49 CFR 173.88.

(b) A solid waste that exhibits the characteristic of reactivity has the EPA Hazardous Waste Number of D003.

[45 FR 33119, May 19, 1980, as amended at 55 FR 22684, June 1, 1990]

§ 261.24 Toxicity characteristic.

(a) A solid waste exhibits the characteristic of toxicity if, using the test methods described in Appendix II or equivalent methods approved by the Administrator under the procedures set forth in §§ 260.20 and 260.21, the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 at the concentration equal to or greater than the respective value given in that Table. Where the waste contains less than 0.5 percent filterable solids, the waste itself, after filtering using the methodology outlined in Appendix II, is considered to be the extract for the purpose of this section.

(b) A solid waste that exhibits the characteristic of toxicity has the EPA Hazardous Waste Number specified in Table I which corresponds to the toxic contaminant causing it to be hazardous.

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TABLE 1.—MAXIMUM CONCENTRATION OF CONTAMINANTS FOR THE TOXICITY CHARACTERISTIC

EPA HW No. ¹	Contaminant	CAS No. ²	Regulatory Level (mg/L)
D004	Arsenic	7440-38-2	5.0
D005	Barium	7440-39-3	100.0
D016	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-8	1.0
D019	Carbon tetrachloride	56-23-5	0.5
D020	Chlordane	57-74-9	0.03
D021	Chlorobenzene	108-90-7	100.0
D022	Chloroform	67-68-3	8.0
D007	Chromium	7440-47-3	5.0
D023	o-Cresol	95-48-7	*200.0
D024	m-Cresol	108-39-4	*200.0
D025	p-Cresol	106-44-5	*200.0
D026	Cresol		*200.0
D018	2,4-D	94-75-7	10.0
D027	1,4-Dichlorobenzene	106-46-7	7.5
D028	1,2-Dichloroethane	107-06-2	0.5
D029	1,1-Dichloroethylene	75-35-4	0.7
D030	2,4-Dinitrotoluene	121-14-2	*0.13
D012	Endrin	72-20-8	0.02
D031	Heptachlor (and its epoxide)	76-44-8	0.006
D032	Hexachlorobenzene	118-74-1	*0.13
D033	Hexachlorobutadiene	87-68-3	0.5
D034	Hexachloroethane	67-72-1	3.0
D008	Lead	7439-92-1	5.0
D013	Lindane	58-89-9	0.4
D009	Mercury	7439-97-6	0.2
D014	Methoxychlor	72-43-5	10.0
D035	Methyl ethyl ketone	78-93-3	200.0
D036	Nitrobenzene	98-95-3	2.0
D037	Pentachlorophenol	87-88-5	100.0
D038	Pyridine	110-86-1	*5.0
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0
D039	Tetrachloroethylene	127-18-4	0.7
D015	Toxaphene	8001-35-2	0.5
D040	Trichloroethylene	79-01-6	0.5
D041	2,4,5-Trichlorophenol	95-95-4	400.0
D042	2,4,6-Trichlorophenol	88-06-2	2.0
D017	2,4,5-TP (Silver)	83-72-1	1.0
D043	Vinyl chloride	75-01-4	0.2

¹ Hazardous waste number.

² Chemical abstracts service number.

* Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

* If o-, m-, and p-Cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/L.

[55 FR 11862, Mar. 29, 1990, as amended at 55 FR 22684, June 1, 1990; 55 FR 26987, June 29, 1990]

EFFECTIVE DATE NOTE: At 55 FR 11862, Mar. 29, 1990, § 261.24 was revised, effective September 25, 1990. At 55 FR 26987, June 29, 1990, the entry for "Heptachlor" in Table 1 was corrected, effective September 25, 1990. For the convenience of the user, the superseded text is set forth below:

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§ 261.24 Characteristic of EP toxicity.

(a) A solid waste exhibits the characteristic of EP toxicity if, using the test methods described in Appendix II or equivalent methods approved by the Administrator under the procedures set forth in §§ 260.20 and 260.21, the extract from a representative sample of the waste contains any of the contaminants listed in Table I at a concentration equal to or greater than the respective value given in that Table. Where the waste contains less than 0.5 percent filterable solids, the waste itself, after filtering, is considered to be the extract for the purposes of this section.

(b) A solid waste that exhibits the characteristic of EP toxicity, but is not listed as a hazardous waste in Subpart D, has the EPA Hazardous Waste Number specified in Table I which corresponds to the toxic contaminant causing it to be hazardous.

TABLE I.—MAXIMUM CONCENTRATION OF CONTAMINANTS FOR CHARACTERISTIC OF EP TOXICITY

EPA hazardous waste number	Contaminant	Maximum concentration (milligrams per liter)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin (1,2,3,4,10,10-hexachloro-1,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo-endo-5,8-dimethano-naphthalene)	0.02
D013	Lindane (1,2,3,4,5,6-hexachlorocyclohexane, gamma isomer)	0.4
D014	Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0
D015	Toxaphene (C ₁₂ H ₈ Cl ₆ , Technical chlordane camphene, 67-68 percent chlorine)	0.5
D016	2,4-D (2,4-Dichlorophenoxyacetic acid)	10.0
D017	2,4,5-TP Silver (2,4,5-Trichlorophenoxypropionic acid)	1.0

Subpart D—Lists of Hazardous Wastes

§ 261.30 General.

(a) A solid waste is a hazardous

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unless it has been excluded from this list under §§ 260.20 and 260.22.

(b) The Administrator will indicate his basis for listing the classes or types of wastes listed in this subpart by employing one or more of the following Hazard Codes:

Ignitable Waste..... (I)
Corrosive Waste..... (C)
Reactive Waste..... (R)
Toxicity Characteristic Waste..... (E)
Acute Hazardous Waste..... (H)
Toxic Waste..... (T)

Appendix VII identifies the constituent which caused the Administrator to list the waste as a Toxicity Characteristic Waste (E) or Toxic Waste (T) in §§ 261.31 and 261.32.

(c) Each hazardous waste listed in this subpart is assigned an EPA Hazardous Waste Number which precedes the name of the waste. This number must be used in complying with the notification requirements of Section 3010 of the Act and certain record keeping and reporting requirements under Parts 262 through 265, 268, and Part 270 of this chapter.

(d) The following hazardous wastes listed in § 261.31 or § 261.32 are subject to the exclusion limits for acutely hazardous wastes established in § 261.5: EPA Hazardous Wastes Nos. F020, F021, F022, F023, F026, and F027.

[45 FR 33119, May 19, 1980, as amended at 48 FR 14294, Apr. 1, 1983; 50 FR 2000, Jan. 14, 1985; 51 FR 40636, Nov. 7, 1986; 55 FR 11863, Mar. 29, 1990]

EFFECTIVE DATE NOTE: At 55 FR 11863, Mar. 29, 1990, § 261.30 paragraph (b) was revised, effective September 25, 1990. For the convenience of the user, the superseded text is set forth below:

§ 261.30 General.

(b) The Administrator will indicate his basis for listing the classes or types of wastes listed in this Subpart by employing one or more of the following Hazard Codes:

Ignitable Waste..... (I)
Corrosive Waste..... (C)
Reactive Waste..... (R)

TARGET COMPOUND LIST (TCL/HSL/SUPERFUND)
ACID-EXTRACTABLES BY GC/MASS
EPA METHODS 625-CLP-M/625/8250/8270
SPECTROPHOTOMETRY MATRIX: WATER/SOIL/SEMISOLID

-
- | | |
|----------|----------------------------|
| _____1. | 2,4,5-Trichlorophenol |
| _____2. | 2,4,6-Trichlorophenol |
| _____3. | 2,4-Dichlorophenol |
| _____4. | 2,4-Dimethylphenol |
| _____5. | 2,4-Dinitrophenol |
| _____6. | 2-Chlorophenol |
| _____7. | 2-Methylphenol |
| _____8. | 2-Nitrophenol |
| _____9. | 4,6-Dinitro-2-methylphenol |
| _____10. | 4-Chloro-3-methylphenol |
| _____11. | 4-Methylphenol |
| _____12. | 4-Nitrophenol |
| _____13. | Benzoic acid |
| _____14. | Pentachlorophenol Phenol |
-

APPENDIX C

DIVE PLAN

**[NOTE: THE DIVE PLAN WILL BE SUBMITTED FOLLOWING THE SELECTION OF
THE USEPA-APPROVED DIVE CONTRACTOR.]**

EXHIBIT D

TARGET COMPOUND LIST: VOCs AND ABNs

TARGET COMPOUND LIST (TCL/HSL/SUPERFUND)
VOLATILE ORGANIC COMPOUNDS
EPA METHODS 624-CLP-M/624/8240/8260
MATRIX: WATER/SOIL/SEMISOLID

- =====
- | | |
|-----|----------------------------|
| 1. | 1,1,1-Trichloroethane |
| 2. | 1,1,2,2-Tetrachloroethane |
| 3. | 1,1,2-Trichloroethane |
| 4. | 1,1-Dichloroethane |
| 5. | 1,1-Dichloroethene |
| 6. | 1,2-Dichloroethane |
| 7. | 1,2-Dichloroethene (total) |
| 8. | 1,2-Dichloropropane |
| 9. | 2-Butanone |
| 10. | 2-Hexanone |
| 11. | 4-Methyl-2-pentanone |
| 12. | Acetone |
| 13. | Benzene |
| 14. | Bromodichloromethane |
| 15. | Bromoform |
| 16. | Bromomethane |
| 17. | Carbon Disulfide |
| 18. | Carbon tetrachloride |
| 19. | Chlorobenzene |
| 20. | Chloroethane |
| 21. | Chloroform |
| 22. | Chloromethane |
| 23. | cis-1,3-Dichloropropene |
| 24. | Dibromochloromethane |
| 25. | Ethyl Benzene |
| 26. | Methylene chloride |
| 27. | Styrene |
| 28. | Tetrachloroethene |
| 29. | Toluene |
| 30. | trans-1,3-Dichloropropene |
| 31. | Trichloroethene |
| 32. | Vinyl Acetate |
| 33. | Vinyl chloride |
| 34. | Xylenes (total) |
- =====

TARGET COMPOUND LIST (TCL/HSL/SUPERFUND)
BASE/NEUTRAL EXTRACTABLES
EPA METHODS 625-CLP-M/625/8250/8270
BY GC/MASS SPECTROPHOTOMETRY
MATRIX: WATER/SOIL/SEMISOLID

- =====
1. 1,2,4-Trichlorobenzene
 2. 1,2-Dichlorobenzene
 3. 1,3-Dichlorobenzene
 4. 1,4-Dichlorobenzene
 5. 2,4-Dinitrotoluene
 6. 2,6-Dinitrotoluene
 7. 2-Chloronaphthalene
 8. 2-Methylnaphthalene
 9. 2-Nitroaniline
 10. 3,3'-Dichlorobenzidine
 11. 3-Nitroaniline
 12. 4-Bromophenyl-phenylether
 13. 4-Chloroaniline
 14. 4-Chlorophenyl phenyl ether
 15. 4-Nitroaniline
 16. Acenaphthylene
 17. Acenaphthene
 18. Anthracene
 19. Benzo(a)anthracene
 20. Benzo(a)pyrene
 21. Benzo(b)fluoranthene
 22. Benzo(g,h,i)perylene
 23. Benzo(k)fluoranthene
 24. Benzyl alcohol
 25. bis(2-Chloroethoxy) methane
 26. bis(2-Chloroethyl) ether
 27. bis(2-Chloroisopropyl) ether
 28. bis(2-Ethylhexyl)phthalate
 29. Butylbenzylphthalate
 30. Chrysene
 31. Dibenzofuran
 32. Dibenz(a,h)anthracene
 33. Diethylphthalate
 34. Dimethylphthalate
 35. Di-n-butylphthalate
 36. Di-n-octylphthalate
 37. Fluoranthene
 38. Fluorene
 39. Hexachlorobenzene
 40. Hexachlorobutadiene
 41. Hexachlorocyclopentadiene
 42. Hexachloroethane
 43. Indeno(1,2,3-c,d)pyrene
 44. Isophorone
 45. Naphthalene
 46. Nitrobenzene
 47. N-nitrosodiphenylamine
 48. N-Nitroso-di-n-dipropylamine
 49. Phenanthrene
 50. Pyrene